

**COMPARATIVE EVALUATION OF SHEAR BOND  
STRENGTH OF THE PORCELAIN LAMINATES USING  
DIFFERENT SURFACE TREATMENTS: AN IN-VITRO  
STUDY.**

*A Dissertation Submitted to the  
Tamil Nadu Dr. M.G.R. Medical University*



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*In partial fulfillment of the requirement for the degree of*

*MASTER OF DENTAL SURGERY*

*(PART II BRANCH I)  
(PROSTHODONTICS AND CROWN & BRIDGE)*

**APRIL 2013**

## **CERTIFICATE**

This is to certify that the dissertation titled “**A COMPARATIVE EVALUATION OF SHEAR BOND STRENGTH OF THE PORCELAIN LAMINATES USING DIFFERENT SURFACE TREATMENTS: AN IN-VITRO STUDY**” is a bonafide record of work carried out by **Dr. LATA S MUSHANNAVAR**, during the period of 2010-2013. This dissertation is submitted in partial fulfillment, for the degree of Master of Dental Surgery awarded by “**Tamil Nadu Dr. MGR Medical University, Chennai**” in the branch of Prosthodontics. It has not been submitted partially or fully for the award of any other degree or diploma.

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## **DECLARATION**

I, **Dr. LATA S. MUSHANNAVAR**, do hereby declare that the dissertation titled “**Comparative Evaluation of Shear Bond Strength of the Porcelain Laminates Using Different Surface Treatments: An In-Vitro Study**” was done in the Department Of Prosthodontics, Tamil Nadu Government Dental College & Hospital, Chennai 600 003. I have utilized the facilities provided in the Government Dental College for the study in partial fulfilment of the requirements for the degree of **Master of Dental Surgery** in the speciality of **Prosthodontics and Crown & Bridge (Branch I)** during the course period **2010-2013** under the conceptualization and guidance of my dissertation guide, **Dr. C. SABARIGIRINATHAN, MDS.**

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Whereas the PG student as part of her curriculum undertakes to research on “**COMPARATIVE EVALUATION OF SHEAR BOND STRENGTH OF THE PORCELAIN LAMINATES USING DIFFERENT SURFACE TREATMENTS- AN INVITRO STUDY.**” for which purpose the PG Guide shall act as principal investigator and the college shall provide the requisite infrastructure based on availability and also provide facility to the PG student as to the extent possible as a Co-investigator.

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**PG student**

Witness

**PG Guide**

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## ABSTRACT

**Background and Objectives:** Porcelain laminate veneers and dentine bonded crowns rely on the synergistic bonding achieved between the porcelain and resin cement in order to survive the rigours of the oral environment. The enhancement of bonding through modification of the internal porcelain surface is advocated in order to increase the intimacy of the bond and may be achieved by exposing the porcelain surface to various treatments. Therefore this study was designed to assess the most effective way of surface treatment of porcelain laminates.

**Methods:** Forty samples of porcelain laminates (IPS Empress 2) were fabricated and randomly assigned three treatment conditions: (i) Group A: no surface treatment and were used as controls, (ii) Group B: etched with 1.23% of APF gel for 10 minutes, (iii) Group C: sandblasted with 50  $\mu$ m Aluminium oxide, (iv) Group D: etched with 5% hydrofluoric acid for 20 seconds. All samples were silanated before cementation with Variolink N resin luting agent. The shear bond strength values were measured in a universal testing machine with a cross-head speed of 1.0 mm/min. Data were compared by one-way ANOVA and Tukey's test at 5% significance level.

**Results:** Surface treatment with 5% HF exhibited the highest mean shear bond strength ( $22.451 \pm 2.710$  MPa) followed by sandblasting ( $15.659 \pm 3.569$ ), APF gel ( $13.025 \pm 1.618$ ) and control ( $10.60 \pm 1.384$ ) group. There was a significant difference between 5% HF ( $p < 0.001$ ) and other groups.

**Interpretation and Conclusion:** Within the limitations of this study, it may be concluded that etching with 5 % HF produced favorable micromechanical retention.

**Keywords:** - *porcelain laminates, surface treatments, silanization*



# ***Introduction***

---

Beauty of smile and esthetics of anterior teeth are considered synonymous. Any change in morphology and shade in relation to anterior teeth invites careful intervention of Prosthodontic treatment.<sup>1</sup>

The search for ideal restorative material to fulfill both esthetics and strength was going on for several centuries. So many restorative materials of both metallic and tooth colored have come into the existence, but the selection of suitable material to the pertaining clinical situation will bring about the success and patient satisfaction.<sup>1</sup>

Dental restorations using all-ceramic materials have become popular in the last couple of decades, primarily because of esthetic properties such as translucency, fluorescence and opalescence that better simulate the appearance of natural dentition. With addition, they also have chemical stability, a coefficient of thermal expansion similar to dentin, biocompatibility, high compressive strength, long term color stability, wear-resistance and their ability to be formed into desired shapes.<sup>1,2</sup>

Ceramic veneers introduced in the early 1980's as cosmetic restorations as opposed to metal-ceramic or all-ceramic full-coverage crowns is a minimal invasive treatment option in reconstructive dentistry. They have gained wide acceptance with dentists and patients and their popularity has been attributed mainly to their esthetic quality, fracture resistance, tissue acceptance, low debond rate and patient satisfaction.<sup>3,4</sup>

A porcelain laminate veneer is an extremely thin shell of porcelain applied directly to the tooth structure. Porcelain laminate veneers have become a popular method of closing diastemas, restoring fractured and malaligned or worn teeth. They were also widely used in cases of enamel hypoplasia, tooth discoloration and intrinsic stains.<sup>2,5</sup>

Tooth preparation is minimal and thus cementation process is vital for the clinical success of ceramic restorations. Ceramic restorations may be cemented with zinc phosphate, glass ionomer, or resin composite cements. When zinc phosphate or glass ionomer cements are used, adequate retention form is necessary. When this is compromised, resin luting systems are recommended.<sup>2,5</sup> Resin luting materials are a mix of monofunctional monomers with a variable amount of fillers of varying sizes, forms and composition. The amount of filler is reduced in comparison to restorative resins, in order to decrease viscosity and allow better adaptation of a rigid restoration to a cavity surface.<sup>6</sup>

A number of theories were put forward in order to explain the strengthening mechanism of these resin cements, but not a single theory is universally accepted. The bond of the resin luting agent to the tooth structure is enhanced by acid etching the tooth structure and by the use of a dental adhesive. The penetration of monomers into a demineralized dentin matrix, followed by polymerization, promotes a micromechanical bond via hybrid layer formation.<sup>2</sup>

Previous investigations revealed that most clinical failures initiate from the cementation or internal surface. This result is consistent with the discussed fracture-surface observations. In a finite element study, the effect of internal surface flows and cement voids in raising internal stresses was shown, this could be critical in determining the mode of clinical failure observed for glass-ceramic crowns. The failures originated from cementation surfaces identified on the internal surfaces as the location of highest tensile stresses and/or critical flaws; this is therefore the surfaces that need to be strengthened.<sup>7</sup>

Based on the concept of “Surface Strengthening”, hydrofluoric acid or Stripit solution was introduced in 1983 for etching porcelain laminate veneer restoration.

Since then, a lot of studies with different approaches like sandblasting, silane treatment, combination of acid etching and silane treatment were advocated for better bonding between porcelain to resin cement.<sup>7,8,9</sup>

Etching ceramics with hydrofluoric acid, ammonium bifluoride, or acidulated phosphate fluoride gel creates a sufficient resin bond that is enhanced with a silane coating of the etched ceramics. Different surface treatments on ceramic surface have been recommended to enhance this adhesion etching with different acids and grinding with diamond burs. All of these procedures are intended to improve the bond strength by producing micromechanical retention and thus modifying the porcelain surface texture.<sup>7,8,9</sup>

In addition to this mechanically retentive surface, the use of silane provides a chemical interaction, which is attributed to its bifunctional characteristics. A high proportion of porcelain's allows reaction of the silane agent both to the crystal portion of the treated porcelain and to the organic portion of the luting agent.<sup>9</sup>

The application of a silane between ceramic and resin composite provides an effective chemical bonding. Etching and silanization increase the surface energy and the wettability of the ceramic substrate. Bonding is usually obtained by the micromechanical retention provided by the acid-etching of the ceramic surface and chemical coupling by the application of a silane coupling agent.<sup>9,10</sup>

At the tooth surface, an adhesive system is used to bond the luting agent to the tooth substrate. Development in bonding technology for dentistry is rapid and a number of new materials are available today.<sup>11</sup>

Since the introduction of the new generation of enamel-dentin bonding systems, the simultaneous conditioning of the substrates (total etch technique) was mainly performed with phosphoric acid following a 3 step clinical procedure. Further



simplification of the bonding procedures lead to reduced steps in the clinical technique combining at least two of the three bonding steps together.<sup>11,12</sup>

Porcelain laminate veneers and dentine bonded crowns rely on the synergistic bonding achieved between the porcelain and resin cement in order to survive the rigours of the oral environment. To achieve this porcelain-resin bonding, optimal surface preparation techniques for chemical and/or mechanical bonding to porcelain substrates are crucial. This in total is done to achieve clinical success when placing indirect porcelain restorations and, when required, repairing them intraorally.<sup>13,14</sup>

Clinicians are often confused regarding the most effective way to treat the intaglio surfaces of indirect porcelain restorations prior to placement with various adhesives and luting resins. They are often equally perplexed about the “ideal” surface treatment for the intraoral repair of preexisting porcelain restorations. This is not surprising, as there appears to be no clear consensus in the dental literature, among “opinion leaders,” or from dental manufacturers on exactly what the optimal surface treatment should, in fact, be. Dental laboratory technicians also appear to lack standardized protocols on how they should treat the surface of finished porcelain.<sup>14</sup>

There is clear lack of consistent guidelines regarding the surface treatment of porcelains as several investigators have advocated numerous modalities with varying concentrations often leading to crowded and unclear concepts raising several significant questions. So, hereby we are making an attempt to address some of these questions, provide some useful general guidelines regarding the management of surface treatment of porcelain restorations by comparing different types of surface treatments with resin cement to ultimately achieve a successful long standing clinical success.



# *Aims and Objectives*

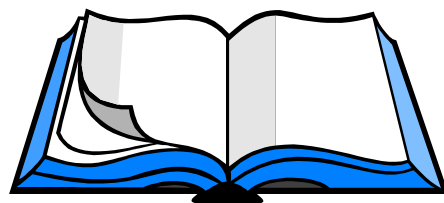
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### **AIMS AND OBJECTIVES OF THE STUDY:**

- i) The **aim** of this study was to evaluate the effect of different ceramic surface pre-treatments on the shear bond strength between porcelain laminates, resin cement and human teeth, with the application of a silane coupling agent.
- ii) The **objectives** of this in vitro study are:  
  
To compare and evaluate the effect of different ceramic surface pre-treatments on the shear bond strength of porcelain laminates with resin cements to human teeth.

### **THE NULL / WORKING HYPOTHESIS:**

- (1) Simplifying the cementation procedure without the application of surface pre-treatment procedure affect the effectiveness of the bond to adhesive resins.
- (2) Differences exist in long-term durability to surface pre-treatments between the adhesive resins with the human teeth.



# ***Review of Literature***

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**Yen TW, Blackman RB, Baez RJ (1993)**<sup>15</sup>evaluated the effect of acid etching on the flexural strength of a feldspathic porcelain and a castable glass ceramic. Fifty specimens of each ceramic material were subjected to five different etch times. A silane coupling agent and composite resin cement were applied. Specimens were examined under scanning electron microscope to elucidate more information on the effect and the depth of etch. There was no significant difference in the mean flexural strengths between the etched and non-etched groups and no significant difference between the different etching times for either material. So they concluded that chemical etching can improve the retention of ceramic laminate veneers without significant loss of strength.

**Kern M, Thompson VP (1994)**<sup>16</sup> investigated the volume loss, morphology, and changes in the surface composition of glass-infiltrated alumina ceramic after sandblasting and silica coating. They stated that etching of dental ceramics with hydrofluoric acid or ammonium hydrogen difluoride is a common procedure to achieve a clean micro retentive surface before bonding or repairing of ceramic restorations. The mean volume loss on various dental ceramics through etching has been reported to be only 1 to 2 $\mu$ m after 1 minute of etching with etching gels. Roughening of ceramic materials by sandblasting has also been used as a substitute for etching. It has the potential to remove significant amounts of material and could affect the clinical fit of the restoration. There are two systems namely; Rocatec / tribochemical system which applies two steps and another is the Silicoater MD system. The authors were of the opinion that in feldspathic ceramics like Empress the volume loss by sandblasting would be excessive because a mere 1 second of sandblasting removed almost 1mm<sup>3</sup> of ceramic material. They concluded that,

sandblasting of feldspathic materials, such as IPS Empress ceramic should be avoided because of an abrasion rate that was 36 times higher.

**Kern M, Thompson VP (1995)<sup>17</sup>** studied the bonding to glass infiltrated alumina ceramic: Adhesive methods and their durability. The tensile bond strength of six bonding systems to In-Ceram ceramic was tested after up to 150 days of storage in isotonic artificial saliva solution and thermal cycling. Sandblasting alone or additional use of a silane did not result in a durable bond of a conventional BIS-GMA composite resin to In-Ceram ceramic. A durable bond was achieved with a combination of tribochemical silica coating and conventional BIS-GMA composite resin or with the combination of sandblasting and a composite resin modified with a phosphate monomer. A delayed degradation in bond strength was recorded for the combination of thermal silica coating and a conventional BIS-GMA composite resin; no reduction in bond strength was found after 30 days, but there was a pronounced decrease in strength after 150 days. This degradation indicated that extended storage in a wet environment was needed in laboratory tests to evaluate the durability of chemical bonds.

**Uno S, Stean H, Finger WJ (1997)<sup>18</sup>** in detail studied about the adhesive bonding of porcelain laminate veneers. In their study, the bonding surfaces of 40 porcelain disks were etched with 13% hydrofluoric acid solution for 8 minutes. Then the etched surface was washed with 70% ethanol and then silane-treated with Scotch-prime. They stated that acid etching and silane treatment have generated high bond strength at the porcelain interface.

**Chang JC, Nguyen T, Duong JH, Ladd GD (1998)<sup>3</sup>** investigated the tensile bond strengths of dual-cured cements between a glass-ceramic and enamel. The investigators used Ceram etch (Gresco products, Inc, Stafford, Texas; hydrofluoric

acid) material for 5 minutes, silane coupling agent (Monobond S) for 60 seconds, Silicoup material A and B, K etchant gel (J. Mortia; 40% phosphoric acid) for 5 seconds. The study compared the tensile bond strengths between Dicor castable ceramics and enamel of four dual-cure cements.

**Olorunfemi BO (1998)<sup>19</sup>** studied the effect of “The Porcelain Fitting Surface on The Porcelain to Composite Bond Strength”. Surface treatments like grit blasting, hydrofluoric acid (10% for 9 min.) etching and silane priming were performed for polished and refractory group of porcelain. They concluded that, combination of the three surface treatments on one disc surface produced highly significant increases in bond strengths at  $p < 0.001$ . Acid etching and silane application are recommended for porcelain fitting surface treatment during the bonding of ceramic veneers, inlays and onlays to the tooth surface.

**Kamada K, Yoshida K, Atsuta M (1998)<sup>20</sup>** studied the effect of ceramic surface treatments on the bond of four resin luting agents to a ceramic material. They performed four ceramic surface treatments to Cerec 2 ceramic material. The group 1-abrasion with No. 600 silicon carbide paper, Group 2-etching with phosphoric acid gel, Group 3-application with silane, Group 4-combination of phosphoric acid gel and silane. They concluded that combined surface treatment of etching with phosphoric acid and application of silane coupling agent provides the highest bond strengths of resin luting agents to Cerec 2 ceramic material after thermal cycling.

**Magne P, Douglas WH (1999)<sup>21</sup>** studied dentin bonding optimization and biomimetic recovery of the crown among porcelain veneers. The investigators used 10% ammonium bifluoride gel for 90 seconds to etch porcelain veneers. Then these dentin bonded porcelain veneers were assessed using functional and cyclic thermal loads with respect to two parameters: coronal stiffness and morphology of the tooth-

restoration interface with two different application modes of same dentin bonding agent.

**Braga RR, Ballester RY, Daronch M (2000)**<sup>22</sup> evaluated the influence of time and adhesive system on the extrusion shear strength between feldspathic porcelain and bovine dentin. In their study, they used 4% hydrofluoric acid to etch the Porcelain truncated cones and then silanized. They did the extrusion shear test after 15 min, 4 h, 12 h, 24 h, and 7 days. The values found at 24 h or 7 days were higher than at 15 min.

**Peumans M, Meerbeek BV, Lambrechts P, Vanherle G (2000)**<sup>23</sup> reviewed the literature about the porcelain veneers. In their review, they stated that, Simonsen and Calamia as well as Horn reactivated the interest in porcelain veneers by introducing special acid etching procedures that substantially improved the long term porcelain veneer retention. They demonstrated that the bond strength of a hydrofluoric acid-etched and silanated veneer to the luting resin composite is routinely greater than the bond strength of the same luting resin to the etched enamel surface. They were of the opinion that, by etching the inner side of the porcelain veneer with hydrofluoric acid and subsequently silanizing the etched surface, the bond strength of a luting composite to the etched porcelain surface has been measured to be higher than the bond strength of a luting composite to etched enamel and even exceeding the cohesive strength of the porcelain itself. Etching the inner side of the porcelain veneer with hydrofluoric acid creates a retentive etch pattern.

Ultrasonic cleaning of etched porcelain in 95% alcohol, acetone or distilled water is indicated to remove all residual acid and dissolved debris from the surface. Inadequate rinsing after etching the porcelain surface may leave remineralised salts, which can be recognized as a white residue or deposit.



Silanization of etched porcelain with a bi-functional coupling agent provides a chemical link between the luting resin composite and porcelain. The bond strength of resin composite to a pre-treated ceramic restoration has been described to be negatively influenced by external factors like water absorption, thermocycling and fatigue. Contamination of the pre-treated surface with die stone, latex gloves, saliva, silicone-based fit-checker paste, and try-in paste will also lower the bond strength.

**Hahn P, Gustav M, Hellwig E (2000)**<sup>24</sup> assessed the strength of porcelain veneers dependent on tooth penetration in an in-vitro study. They used 10% ammonium hydrogen difluoride to etch porcelain veneers and then were silanized and cemented to mandibular incisors with a low viscous luting composite material. Then they analyzed the influence of the incisal preparation on the loadability of teeth restored with porcelain veneers.

**Sen D, Poyrazoglu E, Tuncelli B, Goller G (2000)**<sup>25</sup> proposed that hydrofluoric acid chemical conditioning did not produce good results for alumina reinforced ceramics, and surface sandblasting can be considered a good alternative for creating a micromechanical adhesion-favorable surface.

**Madani M, Chu FCS, McDonald AV and Smales RJ (2000)**<sup>26</sup> conducted a study on the Effects of surface treatments on shear bond strengths between a resin cement and an alumina core. In this study, they divided 45 test specimens into three groups. Group I specimens were treated with a 9.5% hydrofluoric (HF) acid, group II with a 5% HF acid, and group III were sandblasted. All specimens were coated with a silane coupling agent before cementation with Panavia 21 to sandblasted nickel-chromium rods. As a control, group IV consisted of 8 porcelain (Vitadur Alpha) rods treated with a 5% HF acid and silane. It was found that when using Panavia 21 resin cement and Clearfil silane, sandblasted In-Ceram porcelain specimens produced the

highest mean shear bond strength values. Almost similar shear bond strength values were obtained for Vitadur Alpha porcelain and In-Ceram specimens when etched with a 5% hydrofluoric acid. Therefore, these two surface treatments appear to be the methods of choice for the cementation of ceramic restorations. The shear bond strength values of the In-Ceram specimens decreased with an increase in the HF concentration.

**Özcan M, Alkumru HN, Gemalmaz D (2001)**<sup>7</sup> conducted a research on the effect of surface treatment on the shear bond strength of luting cement to a glass-infiltrated alumina ceramic. Eight samples were used for each experimental group. The samples were randomly assigned three treatment conditions: (1) etching for 90 seconds with 5% hydrofluoric acid gel, (2) sandblasting (110- $\mu$ m Al<sub>2</sub>O<sub>3</sub>), and (3) tribochemical silica coating. All samples were silanated and thermocycled for 5,000 cycles altering between 5 and 55°C with 30-second dwell times. The shear bond strength values were measured in a universal testing machine with a cross-head speed of 1 mm/min. They concluded that luting of ceramic with various resins provided varying degrees of bond strengths that were significantly ( $P < .01$ ) increased by the tribochemical silica-coating system in comparison to acid etching or sandblasting.

**Stewart GP, Jain P, Hodges J (2002)**<sup>27</sup> evaluated immediate and 6 month shear bond strengths between a feldspathic ceramic and 4 different resin cements with the use of 6 different surface-conditioning treatment (sanding with 600 grit silicon carbide paper, micro etching with aluminum oxide, sanding followed by silane application, micro etching followed by silane application, hydrofluoric acid etching, and hydrofluoric acid etching followed by silane application). They concluded that hydrofluoric acid etching followed by silane application produced the best bonds at 24

hours and 6 months with all 4 cements. Auto- and light-polymerized adhesives were associated with higher bond strengths to dentin than dual-polymerized adhesives.

**Lee JY and Im EB (2003)**<sup>8</sup> evaluated the shear bond strength of resin cements bonded to pressable porcelain with various surface treatments and stated that hydrofluoric acid etched and silanated group of porcelain surfaces showed the bond strength of 28.30 Mpa, sandblasted and silanated group of porcelain surfaces showed bond strength of 20.88 Mpa. They also stated that SEM view showed differences between untreated porcelain, sandblasted porcelain, and hydrofluoric acid etched porcelain. They noticed that most of the fractures were of cohesive nature.

**Lee JY (2003)**<sup>28</sup> performed a shear bond strength of resin cement bonded to human uncut enamel, cut enamel, and dentin in vitro. In their study, they stated that Horn (1983) proposed etching porcelain laminate veneer restoration with either hydrofluoric acid or Stripit solution, and it is standard protocol to bond etchable porcelains to teeth. Rochette first advocate the use of silane as a coupling agent, and Lacy et al showed that silane treatment increased almost 5 times higher bond strength than that of acid etched porcelain surface.

**Blatz MB, Sadan A, Kern M (2003)**<sup>29</sup> in their review of resin-ceramic bonding stated that common surface pretreatment options for intaglio surface of ceramics include grinding, abrasion with diamond rotary instruments, airborne particle abrasion with aluminum oxide, acid etching and combinations of any of these methods. Acid etching with solutions of hydrofluoric acid (HF) or ammonium bifluoride can achieve proper surface texture and roughness. They concluded that acid etching with HF solutions between 2.5% and 10% applied for 2 to 3 minutes and subsequent application of a silane coupling agent seem to be most successful.

**Terry DA, Blatz MB (2003)**<sup>30</sup> conducted a study on the surface treatments for tooth-colored restorations. The authors recommended that acid-etching with 4% to 9.8% HF to create surface roughness and the application time depends on the crystalline content of the specific ceramic substrate. A higher crystalline content requires less acid etching time and concentration. A silane coupling agent is then applied to the etched ceramic surface. It is important not to place an excess or thick layer of silane because additional layers of hydrolyzed silane will not bond to the porcelain surface and can result in a less than optimal porcelain bond.

**Spohr AM, Sobrinho LC, Consani S, Sinhoreti MAC, Knowles JC (2003)**<sup>31</sup> conducted a study on the influence of surface conditions and silane agent on the bond of resin to IPS empress 2 ceramic. One hundred twenty samples were made, embedded in resin, and randomly divided into six groups: group 1 = sandblasting (100  $\mu$ m), no silanation; group 2 = sandblasting (100  $\mu$ m), silane treatment; group 3 = sandblasting (50  $\mu$ m), no silanation; group 4 = sandblasting (50  $\mu$ m), silane treatment; group 5 = hydrofluoric acid etching, no silanation; and group 6 = hydrofluoric acid etching, silane treatment. The disks were bonded into pairs with adhesive resin cement. All samples were stored in distilled water at 37°C for 24 hours and then thermocycled. The samples were submitted to tensile testing. They recorded the highest bond strength of 25.6 MPa for group 6, followed by 16.4 MPa for group 5. They concluded that combined application of 10% hydrofluoric acid and silane enhanced the bond strength between the IPS Empress 2 ceramic framework and resin agent.

**Borges GA, Spohr AM, Goes MF, Sobrinho LC and Chan DCN (2003)**<sup>32</sup> studied the effect of etching and airborne particle abrasion on the microstructure of different dental ceramics. Five copings each of IPS Empress, IPS Empress 2,

Cergogold, In-Ceram Alumina, In-Ceram Zirconia, and Procera were fabricated and each coping was longitudinally sectioned into 4 equal parts by a diamond disk. The resulting sections were then randomly divided into 3 groups depending on subsequent surface treatments: Group1, specimens without additional surface treatments (control); Group 2, specimens treated by use of airborne particle abrasion with 50  $\mu$ m aluminum oxide; and Group 3, specimens treated with 10% hydrofluoric acid etching. They concluded that hydrofluoric acid etching and airborne particle abrasion with 50 $\mu$ m aluminum oxide increased the irregularities on the surface of IPS Empress, IPS Empress 2 and Cergogold ceramics. Hydrofluoric acid and airborne particle abrasion with 50 $\mu$ m aluminum oxide did not change morphologic microstructure on the surface of In-Ceram Alumina, In-Ceram Zirconia and Procera.

**Nagai T, Kawamoto Y (2004)**<sup>33</sup> studied the Effect of hydrofluoric acid etching on bond strength of composite luting agent to lithium disilicate ceramic material. In their study, two sizes of disk specimens were made from a lithium disilicate-based ceramics (IPS Empress 2) and their surfaces were separately prepared with three methods: etching with phosphoric acid (PE), etching with hydrofluoric acid (HF), and air-borne particle abrasion with alumina (AA). Each group was further divided into two sub-groups: bonding with the Panavia F material (PF), and silane treatment followed by bonding with the Panavia F material. Shear testing was performed both before and after 20,000 thermocycles. They concluded that, hydrofluoric acid etching effectively enhanced bond strength of the Panavia luting agent to the ceramic material, regardless of the application of silane primer.

**Begazo CC, De Boer HD, Kleverlaan CJ, van Waas MAJ, Feilzer AJ (2004)**<sup>34</sup> found the optimal choice of luting cement to Synthoceram, an aluminum oxide-reinforced glass ceramic material. The bond strength of five different

commercial luting cements (Ketac Cem, Rely XLuting, Fuji Plus, Panavia F & Xeno Cem.) to the ceramic material was evaluated. The effect of surface treatments, etching, sandblasting, silanizing, and a combination of these treatments was also investigated. Based on the results of this study, they concluded the use of resin composite based cements is preferred for cementation of an all-ceramic restoration with an aluminum oxide reinforced glass ceramic base. Surface treatments of etching and/or sandblasting followed by silanization provide the highest bond strength values.

**Zhang Y, Lawn BR, Rekow ED, Thompson VP (2004)**<sup>35</sup> studied the effect of sandblasting on the long-term performance of alumina and zirconia dental ceramics. Specimens with polished surfaces are used as a control. Tests are conducted with monotonically increasing (dynamic) and sinusoidal (cyclic) loading on the spherical indenter, up to the point of initiation of a radial fracture at the ceramic bottom surface immediately below the contact. Strengths of sandblasted specimens showed significant reductions in both dynamic and cyclic tests, indicative of larger starting flaws. They concluded that surface abrasion treatments can be an important degrading factor in long-term performance of all-ceramic crowns.

**Filho AE, Vieira LCC, Ara'ujo TE and Junior SM (2004)**<sup>36</sup> studied The Effect of Different Ceramic Surface Treatments on Resin Microtensile Bond Strength. They formed four groups with Group 1 specimens treated with 9.5% hydrofluoric (HF) acid for 20 seconds and silane (S) for 3 minutes; Group 2: silane for 3 minutes; Group 3: 9.5% HF acid for 20 seconds; Group 4: no treatment. They found the highest bonding strength in Group 1 with 56.8 Mpa, Group 2 with 44.8 Mpa and Group 3 with 35.1 Mpa. They concluded that ceramic silanization was individually the most significant factor responsible for the bond strength, acid etching and silanization

promoted maximum bond strength and sandblasting itself did not provide adequate bond strength.

**Soares CJ, Soares PV, Pereira JC, Fonseca RB (2005)**<sup>37</sup> searched the peer-reviewed articles published in English between 1965 and 2004 in dental journals about the surface treatments like sandblasting, etching techniques and silane coupling techniques. They stated that for the leucite-reinforced ceramics, using hydrofluoric acid for 60 seconds is sufficient. For lithium disilicate ceramics etching with 9.5% hydrofluoric acid showed greater results compared with the 4% acidulated phosphate fluoride. Excessive airborne particle abrasion has induced chipping or a high loss of ceramic material and is therefore not recommended for cementing silica-based all-ceramic restorations.

According to Peumans and colleagues, silane has functional groups that promote chemical bonding with hydrolyzed silicon oxide from the ceramic surface and with the methacrylate group from the adhesive system or the composite cement by copolymerization. Della Bona and colleagues have demonstrated an increase in adhesive resistance when using silane with ceramics reinforced with feldspar, leucite, or lithium disilicate, also concluding that only the application of silane over nontreated ceramics presents a low resistant adhesive interface.

**Bona DA, Anusavice KJ, Mecholsky Jr JJ (2006)**<sup>38</sup> investigated the apparent interfacial fracture toughness of resin/ceramic systems. A hot-pressed leucite-based ceramic (E1; IPS Empress) and a hot-pressed lithia disilicate-based ceramic (E2; IPS Empress 2) were divided into 6 groups and subjected to different surface pretreatments like, Group 1 (E1): 9.5% hydrofluoric acid (HF) for 1 min, rinsed for 30 sec and dried with oil-free air; Group 2(E2): slaine (S) coating for 5 min. and air dried; Group 3(E3): HF for 1 min, rinsed for 30 s, air-dried, followed by

application of S; Group 4 (E2): HF for 1 min; Group 5 (E2): S coating for 5 min; Group 6 (E2): HF and S. All fractures originated from indentation sites. Estimation of interfacial toughness was feasible by fracture mechanics and fractography. The interfacial fracture toughness for the systems was affected by the ceramic microstructure and surface treatment.

**Matsumura H, Aida Y, Ishikawa Y, Tanoue N (2006)**<sup>39</sup> in their case report of Porcelain laminate veneer restorations bonded with a three-liquid silane bonding agent and a dual-activated luting composite advocated the use of ultrasonic cleaning with methanol using a polyethylene cup. Ultrasonic cleaning after hydrofluoric acid etching effectively removes precipitates and acid from the etched surface. Also, the water that penetrated onto the etched surface is probably replaced by methanol, which is more volatile than water. Thus, after air-drying, the wetting ability of the bonding agent may be enhanced in the case of etched and methanol-treated porcelain, compared to the etched and water-sprayed porcelain. The use of polymer cup during the ultrasonic cleaning is beneficial for the porcelain laminate veneer restorations, as it might reduce the possibility of microfracture in thin marginal areas of the brittle porcelain restorations.

**Proenca JP, Erhardt MCG, Valandro LF, Aceves GG, Carmona MVB, Salmeron RDC et al (2006)**<sup>10</sup> studied in detail about the influence of ceramic surface conditioning and resin cements on microtensile bond strength to a glass ceramic. Eighteen samples were divided into two groups. Group 1: no conditioning/control and Group 2: 5% hydrofluoric acid etching for 20 seconds and silanization for 1 minute (HF+SIL). Ceramic blocks were cemented to the composite resin blocks and were stored in humidity at 37<sup>0</sup> C for 7 days and serially sectioned to produce 25 beam specimens per group with a 1.0 mm<sup>2</sup> cross-sectional area. Specimens were



thermaocycled (5000 cycles, 5<sup>0</sup> C-55<sup>0</sup> C) and tested in tension at 1mm/min. They observed a higher bond strength of combined HF + Silane over group 1 and concluded that ceramic surface treatment with hydrofluoric acid and a silane coupling agent has a positive influence on the cementation of the Lithia disilicate based ceramic with autopolymerizing resin based cement.

**Addison O, Marquis PM, Fleming GJP (2006)<sup>13</sup>** studied the impact of hydrofluoric acid surface treatments on the performance of a porcelain laminate restorative material. Sets of 30 Vitadur-Alpha discs were etched with HF acid of three different concentrations (5, 10 and 20%) and for three different etching periods (45, 90 and 180 s). Mean flexure strengths, standard deviations and the associated Weibullmoduli (m) and characteristic stress were determined using biaxial flexure (ball on ring). A significant reduction of the flexural strength of low fusing feldspathic porcelain has been demonstrated to result from etching. As a result it has been proposed that the longevity of porcelain laminate veneer restorations can be improved by adherence to optimizing the pre-cementation HF acid etching time and concentration.

**Zarone F, Sorrentino R, Vaccaro F, Traini T, Russo S, Ferrari M (2006)<sup>40</sup>** conducted a study on acid etching surface treatment of feldspathic, alumina and zirconia ceramics: a micromorphological SEM analysis. 45 specimens of feldspathic, alumina and zirconia ceramics were collected. 5 specimens of each type of ceramics were not subjected to any surface treatment and analyzed with SEM as controls. The remaining 30 samples were etched with 40% hydrofluoric (HF) acid gel for 2 min and then subjected to SEM analysis. They concluded that etching with 40% HF acid for 2 min produced micro-retentions on the surface of feldspathic ceramics, but did not achieve proper surface texture and roughness on both alumina and zirconia ceramics.

**Bitter K, Paris S, Hartwig C, Neumann K and Kielbassa AM (2006)<sup>41</sup>**

tested the shear bond strengths of different substrates bonded to lithium disilicate ceramics. Ceramic cubes were luted either with a one-step or multiple-step total etching bonding system to ground surfaces of human enamel, dentin, and resin core materials. Resin core materials were additionally pretreated with hydrofluoric acid (HF) or were silica-coated (CoJet System). Shear bond strengths were determined after 24-hour water storage (n=10) and thermocycling. Bond strengths to enamel, dentin, and silica-coated composites were significantly higher compared to untreated and HF-pretreated composites. Indeed, silica coating of the composite resins significantly increased the bond strength to ceramics.

**Nagayassu MP, Shintome LK, Uemura ES, Araújo JEJ (2006)<sup>9</sup>**

studied the effect of surface treatment on the shear bond strength of a resin-based cement to porcelain. They divided the 60 porcelain discs into six groups, according to the surface treatment: etching with 10% hydrofluoric acid for 2 or 4min (G1 and G2); 50- $\mu$ m particle aluminum oxide sandblasting for 5 s (G3); sandblasting followed by etching for 2 or 4min (G4 and G5) and control - no treatment (G6). A silane agent was applied to the treated surface of both discs of each pair. Bistite II DC dual-cure resin cement was applied and the B discs were bonded to their respective A discs. Specimens were stored in distilled water at 37°C for 24 h and were tested in shear strength at a crosshead speed of 2 mm/min. Means in MPa were: G1:  $14.21 \pm 4.68$ ; G2:  $8.92 \pm 3.02$ ; G3:  $10.04 \pm 2.37$ ; G4:  $12.74 \pm 5.15$ ; G5:  $10.99 \pm 3.35$ ; G6:  $6.09 \pm 1.84$ . The authors concluded that 2-min hydrofluoric acid etching produced a favorable micromechanical retention that enhanced resin cement bond strength to porcelain.

**Borges GA, Goes MF, Platt JA, Moore K, Menezes FH, Vedovato E (2007)**<sup>2</sup> evaluated the extrusion shear strength between an alumina-based ceramic and three different cements. They used 10% phosphoric acid to etch the porcelain surfaces for 1 minute, dried with compressed oil-free air. Then they evaluated the bond strength between a densely sintered alumina ceramic and bovine dentin with 2 adhesive resin cements and a resin-modified glass ionomer cement using an extrusion shear strength test.

**Conrad HJ, Seong WJ, Pesun IJ (2007)**<sup>42</sup> published a systematic review on current ceramic materials and systems with clinical recommendations. In their review, they were of the opinion that for conventional glass-ceramic restorations, the adhesive technique is critical for successful bonding. Surface treatment of the porcelain by etching with 5% to 9.5% hydrofluoric acid and etching of the tooth structure with 37% phosphoric acid and the application of silane coupling agent provided the highest bond strength of adhesive-resin cement to feldspathic material. Bond strength to etched surfaces is improved by creating deep invaginated spaces where resin can flow and interlock.

They were of the opinion that due to the abrasion rate with subsequent volume loss and changes in morphology, feldspathic restorations should never be airborne-particle abraded to improve the roughness of the internal surface, only acid-etched.

**Vasconcellos WA, Alvim HH, Saad JRC, Susin AH (2007)**<sup>43</sup> studied the effects of surface treatment on the microtensile bond strength of Duceram Plus (DP), IPS Empress 2 (IE) and In Ceram (IC) ceramic materials to dentin by different surface treatments like sandblasting, hydrofluoric acid and tribochemical silica coating in eighteen extracted molars. They observed highest bonding strength in the group of hydrofluoric acid treatment for Duceram Plus (DP) ceramics, whereas IPS Empress 2

ceramics results did not show any significant difference in bond strength for the distinct surface treatments and for the In-Ceram alumina ceramic system, bond strength presented a significant difference for the different surface treatments, the highest values being associated with tribochemical silica coating process, which was significantly higher than those of the other tested conditions.

They concluded that,  $\mu$ TBS is influenced by the interaction between surface treatment and material composition. The bond strength of feldspathic ceramic Duceram Plus is significantly high for hydrofluoric acid etching, while that of ceramic IPS Empress 2 is not significantly different for distinct surface treatments. The bond strength of In-Ceram alumina is significantly high for tribochemical silica coating process.

**Hung CY, Lai YL, Hsieh YL, Chi LY, Lee SY (2008)<sup>44</sup>** evaluated the effects of simulated clinical grinding and subsequent heat treatment on microcrack healing of a lithium disilicate ceramic. Sixty disk specimens of Empress 2 core ceramic were fabricated and divided into six groups as Group 1: control; Group 2: ground with a diamond bur; Group 3: ground with a diamond bur, followed by simulated veneer firing; Group 4: Simulated veneer firing; Group 5: ground with a diamond bur after simulated veneer firing; Group 6: ground with a diamond bur after simulated veneer firing followed by re-glazing. In this study, intaglio grinding of core ceramic with diamond burs was shown to induce detrimental microcracks and result in reductions in flexural strength and reliability.

**Alex G (2008)<sup>14</sup>** in his extensive review paper entitled “Preparing Porcelain Surfaces for Optimal Bonding” tried to address some questions, provided some guidelines and attempted to make some sense of the various methodologies currently advocated for the treatment of porcelain surfaces prior to placement. In his opinion,

HF in 4% to 10% concentration range can be used safely for dental procedures, including intraoral repair, provided caution and common sense are employed. He stated that one recent study showed that a seven to ten minute application of 1.23% APF gel on a leucite containing porcelain (IPS Empress) produced a shear bond strength to composite similar to a four-minute etch with 9.6% HF. According to him, other studies have also shown that etching with APF, even with prolonged application times, results in very shallow etching patterns when compared to HF etching for much shorter time periods.

A study by Barghi showed a 50% leucite content porcelain (Fortress) required a longer etching time (ie, 150 to 180 seconds) with 10% HF when compared to a 27% leucite content ceramic (Ceramco) that only required a 60-second etch with 10% HF. According to this author, it is certainly possible that a difference in leucite concentration, size/orientation of crystals, or microstructure might affect etching times.

The manufacturer of two lithium disilicate-based ceramics (IPS e.max and IPS e.max Press) recommends a very specific etching time of only 20 seconds with 5% HF and studies by Pisani- Proenca J et al and Kim JS supported this. Some opinion leaders/studies recommend 90 seconds of etching with 10% HF for stackable feldspathic porcelains, while others recommend a 120- to 150-second etch with 9.5% HF. The manufacturers of two popular 9.5% HF etching gels (HF etching gel, Bisco and HF etching gel, Ultradent Products) recommend etching times of 90 and 60 seconds respectively.

In an unpublished study, ByoungSuh tested 10 different feldspathic porcelains by HF etching (i.e., 9.5% and 4.0%), followed by contact angle analysis and microscopic evaluation. He first particle-abraded the samples (i.e., sandblasting) and

found they all showed an acceptable microscopic etching pattern after a four- to five-minute etch with 4% HF. These same porcelains showed a similar etching pattern when 9.5% HF was used for 90seconds. The authors of another study found statistically higher composite shear bond strength to several stackable porcelain disks when they were etched with 10% HF for two minutes as opposed to a longer etch for four minutes at the same HF concentration. They attributed the lower bond strength with the longer etching time of four minutes to “over-etching” and actual weakening of the porcelain surface.

Finally, in his fairly extensive review, the author concluded that no single specific HF concentration and application time exists that is optimal for etching all porcelains and made some generalizations regarding porcelain surface treatment. They were,

- a) Low-pressure sandblasting, followed by 60 to 120 seconds of etching with 9% to 10% HF, has scientific support and validity when treating conventional powder/liquid stackable porcelains. A variation that also appears effective is sandblasting followed by 4% to 5% HF applied for four to five minutes.
- b) In the case of IPS Empress ceramic, the recommended treatment protocol of 5% HF applied for 60 seconds has scientific support and should be followed.
- c) High-strength alumina and zirconia core-based crowns cannot be etched with HF. Silica coating, followed by silane application, has been shown to be available alternative.
- d) Proper use of hydrolyzed silane, in conjunction with warm-air drying, has scientific support and is advisable after HF etching of feldspathic porcelains or silica coating of high alumina, zirconia, metal and composite surfaces.

**Aboushelib MN, GhoniemM ,Mirmohammadi H, Salameh Z (2009)<sup>45</sup>**

conducted a full length research on the general principles for achieving adequate bond to all ceramic restorations. In their research, 60 ( Zirconia and glass ceramic 30 each) samples were divided into three groups and surface treated with airborne particle abrasion with 50 mm aluminum oxide particles, HF acid and silane coupling agent and airborne particle abrasion in combination with application of HF acid and silane coupling agent. They concluded that bonding to glass ceramic relies on HF etching and silane treatment.

**Kitayama S, Nikaido T, Takahashi R, Ikeda M, Foxton RM, Sadar A et al**

**(2010)<sup>46</sup>** studied the effect of primer treatment on bonding of resin cements to zirconia ceramic. They used airborne-particle abraded with 70 µm aluminium oxide particles at 0.5 MPa for 5 seconds at a distance of 10 mm. thereafter, all the specimens were ultrasonically cleaned in distilled water for 10 min and air dried. They examined the effect of primer treatment on tensile bond strengths of five resin cements to silica-based and zirconia ceramics. They concluded that, the primers containing a silane coupling agent were effective in improving the bonding of resin cements to silica-based ceramic and primers containing a phosphate ester monomer were effective in improving the bonding of resin cements to zirconia ceramic.

**Mathew CA, Mathew S, Karthik K S (2010)<sup>47</sup>** in their review on ceramic

lamine veneers stated that, it was in 1975 Rochette explained the concept of acid etching porcelain and bonding to the tooth, and described a technique for making ceramic restorations for fractured incisors without operative influence made the pioneers in veneers to turn towards porcelain, one of the most popular and attractive materials in the dental armamentarium. In 1985, John R. Calamina reported a clinical

study in which 200 porcelain laminate veneers were placed, and found that longevity and aesthetics were far more superior to the unetched veneers.

Investigation of shear bond strength of composite resin to porcelain to optimize variable for bonding porcelain laminate veneers was done by Stangel in 1987, which concluded that etching porcelain significantly increased the bond strength. Raymond Lu in 1992 investigated the composite resin to porcelain interface and concluded that surface treatment with hydrochloric acid and/or silane coupling agents improved bonding and fracture strength was altered by resin bonding.

**Zortuk M, Kilic K, Gurbulak AG, Kesim B, Uctasl S (2010)<sup>48</sup>** conducted a study on the tensile bond strength of a lithium-disilicate pressed glass ceramic to dentin with different surface treatments. Sixty 7×3 mm pressed ceramic discs of IPS e.max were fabricated and randomly assigned to six groups of different dentin surface treatments (control, desensitizer, disinfectant, saliva, blood, and hydrogen peroxide). Representative samples of fractured specimens were observed by SEM (scanning electron microscopy). The authors concluded that saliva contamination, blood contamination, and hydrogen peroxide application influenced the tensile bond strengths of adhesive ceramic and adhesive-dentin interfaces, whereas no such detrimental effect was observed with Clinpro white varnish and chlorhexidine gluconate applications.

**Al-Taie LA, Mohammed SA (2010)<sup>49</sup>** studied the effects of different acid porcelain surface treatments on composite - porcelain shear bond strength. Thirty two ceramic fused to metal specimens were prepared, sandblasted with 50 µm aluminum oxide and divided in to four groups of 8 samples: Group I: Etching with 1% hydrofluoric acid solution for 2 minutes .Group II: Etching with 5% hydrofluoric acid solution for 2 minutes. Group III: Etching with 10% hydrofluoric acid solution for 2



minutes. Group IV: Etching with 37% phosphoric acid gel for 15 seconds. Monobond-s, Heliobond. Shear bond strength was determined by a universal testing machine (Zwick 1454) at a cross head speed 5 mm/minutes. They concluded that surface preparation of the ceramic with 10% hydrofluoric acid for two minutes recorded the highest bond strength with the composite, most of specimens treated with hydrofluoric acid exhibited cohesive failure within porcelain.

**Pini NP, Aguiar FHB, Lima DANS, Lovadino JR, Terada RSS, Pascotto RC (2012)<sup>50</sup>** in their review article entitled “Advances in dental veneers: materials, applications and techniques” felt that effective etching of the ceramic surface is considered an essential step for the clinical success. Alteration of the surface topography by etching will result in changes in the surface area and in the wetting behavior of the porcelain. This may also change the ceramic surface energy and its adhesive potential to resin. Differences in ceramic composition will also produce unique topographic changes after etching procedures. The aim of pre-cementation surface modification of the porcelain is to increase the modification of the surface area available for bonding and to create undercuts that increase the strength of the bond to the resin luting cement.

They also felt that, acid conditioning with hydrofluoric acid is efficient in removing superficial defects and rounding off the remaining flaw tips, thereby reducing stress concentrators and increasing the overall strength. Clinical studies have indicated that this protocol significantly increases the expected clinical life span of the restoration. Silanization of etched porcelain with a bifunctional coupling agent provides a chemical link between the luting resin composite and porcelain. A silane group at one end chemically bonds to the hydrolyzed silicon dioxide at the ceramic

surface and a methacrylate group at the other end copolymerize with the adhesive resin.

**Chen L and Suh BI (2012)**<sup>51</sup> reviewed the literature from 2008-2012, focusing on the latest resin bonding techniques (including surface treatment, priming and cementation) for dental all-ceramic materials, especially the two dominant materials zirconia and lithium disilicate ceramics. In their extensive review, they found many investigators stating the interesting facts about surface treatments. According to them, Blatz et al., 2003 and Conrad et al., 2007 stated that the glass ceramics which contains various amounts of glass/silica compositions, such as lithium disilicate, etching with 4-9.5% hydrofluoric acid has been proven a successful surface treatment method to provide surface roughness for mechanical interlocking/bonding.

Nagai et al., 2005; Panah et al., 2008 and Brum et al., 2011 found that among different surface treatments, such as phosphoric or HF acid etching and air-abrasion with alumina, etching with hydrofluoric acid was the most effective in enhancing the bond strength of resin material to lithium-disilicate ceramics. Pollington et al., 2010 stated that lithium disilicate IPS e. max CAD had the highest bond strength when it was the HF-etched after machined, compared to being machined only or machined/grit blasted. Panah et al., 2008; Nagai et al., 2005 stated that silane treatment after HF-etching remains the most effective method for improving resin bonding with silica-based ceramics. Queiroz et al., 2012 stated that the primers containing only silane monomer was the most effective for improving resin bonding to silica-based ceramics.

Finally, the authors of this review concluded that hydrofluoric acid-etching and subsequent silane treatment is the most preferred bonding method.

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# *Methodology*

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This in vitro study was conducted to evaluate and compare the shear bond strength of different ceramic pre-surface treatments of porcelain veneers luted to the tooth structure using resin cement.

The different ceramic pre-surface treatments which are used in this study are having different bonding mechanisms.

The materials used for this study are as follows:-

<b>Sl. No</b>	<b>Material</b>	<b>Manufacturer</b>	<b>Type</b>	<b>Batch no</b>	<b>Chemical composition</b>
<b>1.</b>	Variolink N	Ivoclar Vivadent, Schaan, Liechtenstein	Dual curing/light curing resin cement	Catalyst N01584  Base N01552	BisGMA, UDMA, TEGDMA, barium glass, and silica fillers, YbF3
<b>2.</b>	Variolink N (Excite DSC)	Ivoclar Vivadent, Schaan, Liechtenstein	Bonding agent	M04952  Two-step/etch and rinse	HEMA, DMA, phosphoric acid acrylate, highly dispersed silicon dioxide/ethanol
<b>3.</b>	Monobond-S	Ivoclar Vivadent, Schaan, Liechtenstein	Silane coupling agent	N01595	Alcoholic solution of silane methacrylate

4.	IPS Ceramic etching gel	Ivoclar Vivadent, Schaan, Liechtenstein	Etchant	N39215	5% Hydrofluoric Acid
5.	Professional APF gel	Pascal	Etchant	091114	Acidulated Phosphate Fluoride

BisGMA: Bisphenol-A diglycidyl ether dimethacrylate

EBPADM: Ethoxylated bis-phenol-adimethacrylate

TEGDMA: Triethylene glycol dimethacrylate

UDMA: 7,7,9-trimethyl-4,13-dioxo-3,14-dioxo-5,12-diazahexadecane-1,16-dimethacrylate; YbF<sub>3</sub>: Ytterbium trifluoride

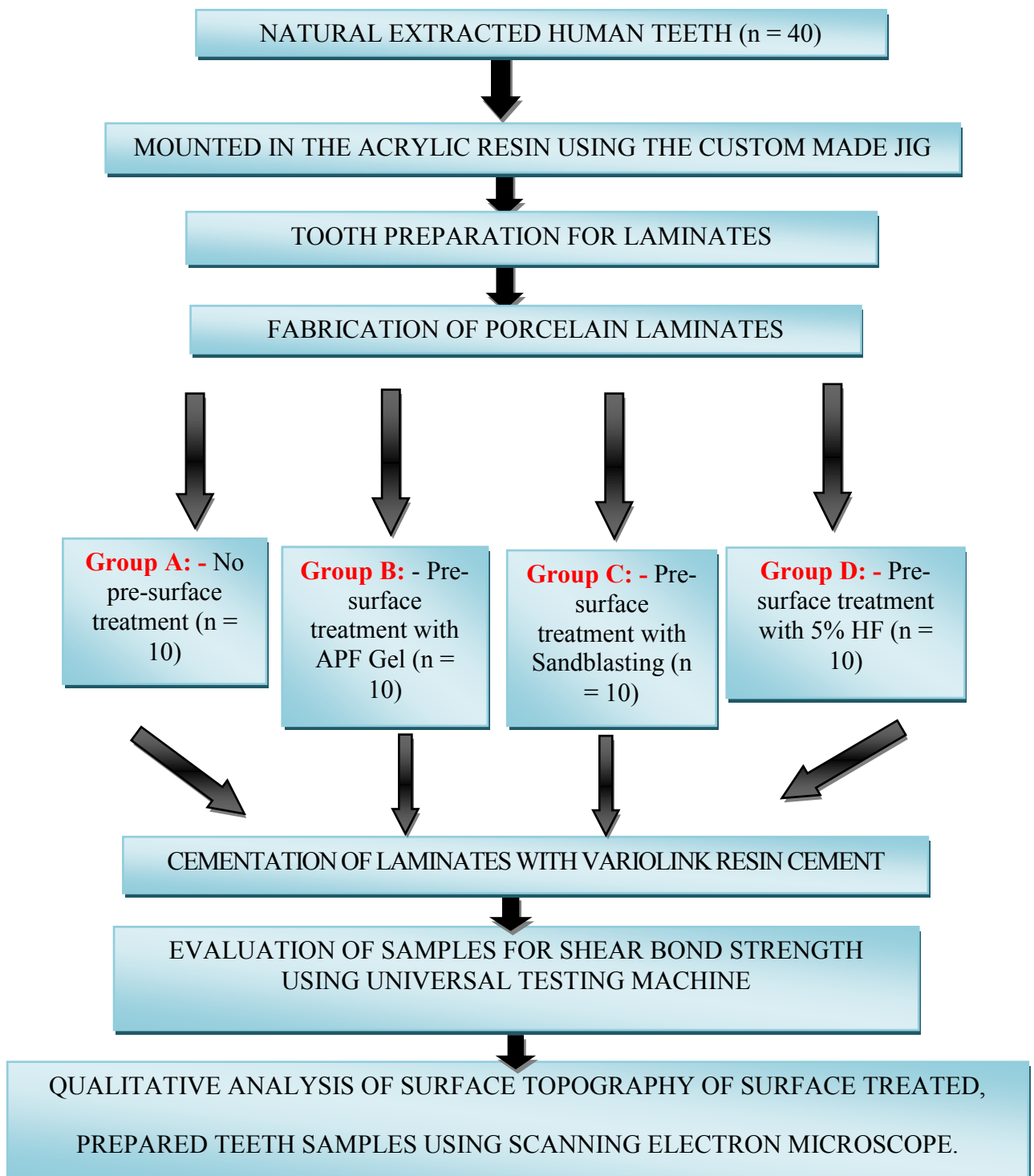
HEMA: 2-hydroxyethylmethacrylate; DMA: aliphatic dimethacrylate;

APF: Acidulated Phosphate Fluoride

### **ARMAMENTARIUM (Fig-1)**

- 1) Natural extracted tooth
- 2) Diamond disc (EDENTA AG, Switzerland)
- 3) Self cure acrylic resin (DPI, India)
- 4) Silicone cup
- 5) Wax knife
- 6) Acrylic trimming burs
- 7) Sandpaper mandral
- 8) Rubber bowl
- 9) Explorer
- 10) Aerotar hand piece (Pana air, NSK, Japan)
- 11) Contra angle hand piece (NSK , Japan)
- 12) Polishing cup
- 13) Electronic Vernier caliper (Digimatic)
- 14) Pumice
- 15) Depth orientation diamond abrasive
- 16) Flat end tapered diamond abrasive
- 17) Fine grit diamond abrasive
- 18) Blade parker blade (No-15)
- 19) Press wax (Delta, India)
- 20) P K Thomas kit (Dispodent)

## STUDY DESIGN:



## **METHODOLOGY:**

I. Tooth Preparation

II. Laminate Fabrication

III. Experimental design- Randomized four groups with each group containing 10 teeth subjected to study

Group A: No surface pre-treatment (Control group)

Group B: Etched with 1.23% APF gel for 10 minutes

Group C: Sandblasted with 50  $\mu$ m Aluminium oxide at 60 psi at 0.5 Mpa for 5 seconds at a distance of 10 mm

Group D: Acid etched with 5% hydrofluoric acid for twenty seconds

IV. Cementation of Veneers to the tooth by Variolink N cement

V. Measurement of Shear Bond Strength by Universal Testing Machine (UTM)

VI. Statistical Evaluation

VII. Qualitative analysis of surface topography of surface treated, prepared teeth samples using scanning electron microscope.



## **I. TOOTH PREPARATION:**

Freshly extracted, non-carious permanent human incisors that were not endodontically treated were selected for this study. Calculus and residual periodontal tissue were removed using a surgical knife, scaler, and curette. All teeth were stored in 0.1% thymol solution at room temperature immediately after extraction. None of the extracted teeth had been stored for longer than 6 months.

After that each tooth was placed in to a silicone mold and embedded in auto-polymerizing methylmethacrylate resin (DPI, Mumbai, India). After hardening the resin in a pressure pot for 10 min, the specimens were wet-ground sequentially to 600-grit using SiC sandpaper, to obtain a flat surface in superficial dentin. The tooth surfaces were kept moist throughout the procedure of specimen preparation.

The facial surfaces of the teeth were prepared to accommodate veneers of equal thickness. Facial reduction was 0.3 mm at the cervical third and 0.5 mm at the middle and incisal thirds. 1 mm of incisal reduction with 1 mm height palatal chamfer was done for incisal and palatal surfaces. Tooth preparations were extended to include inter proximal contacts with rotary instruments and a water coolant. Self limiting depth-cutting disks of 0.3 mm and 0.5 mm were used to define the depth cuts, and then 1.2 mm chamfer diamond burs were selected to refine the preparation. All tooth preparations were completed entirely in enamel, without sharp line angles.

## **II. LAMINATE FABRICATION:**

Once the tooth has been prepared Silicone separating media is applied on the surface. Then Wax pattern is fabricated with Press wax (DELTA) on individual tooth samples so as to closely adapt to the tooth surface with uniform thickness of 0.6 mm and sprued. Ceramic veneers were then pressed after investment. The fabricated Wax

pattern is kept in water for 10 minutes in order to relieve the residual stress. Then the Wax pattern is sprued and invested by phosphate bonded investment using auto mixer machine.

The investment is allowed to set for 45 minutes before keeping in for burn-out furnace (NEY, VULCAN 3-130). The Wax burn out is done at temperature around 930 degree centigrade. Once the temperature reaches the above said degree, the pressable ceramic furnace is started for pre-heating one hour before scheduled pressing. The preheating temperature in the furnace is about 700 degree centigrade. After burn-out is completed, the ring is immediately placed in the pressing machine with ceramic button and plunger. The pressing is started. The overall time period for the Pressing is 30minutes. In the initial 5 minutes there is constant temperature rise of 40 degree centigrade per minute and once the temperature reaches 920 degree centigrade the pressing is started which takes around 21 minutes.

Once the pressing is over the ring is taken out of the machine, and left for bench cooling. Once the pressing is over the ring is taken out of the machine, and left for bench cooling. All procedures were performed with IPS Empress 2 materials and protocol. After divestment, the veneer fit was verified with green aerosol sprayed over the tooth surface. High spots on the ceramic veneers were removed with a diamond medium grit round bur. All ceramic veneers were then reduced to 0.3 mm at the cervical third and 0.5 mm at the incisal two thirds with green stones.

The IPS layering technique was selected for all treated ceramic veneers. Ceramic was cutback before both a wash firing and application of enamel ceramic and relative firing cycles were performed. The amount of cutback was 0.2 mm facially at the incisal two thirds, and 0.5 mm incisally. Enamel ceramic was then applied and finished with diamond burs. Their dimensions were standardized again after

measurement with an electronic Vernier caliper (DIGIMATIC) for the height and a thickness. Final dimensions of all ceramic veneers were 0.3 mm and 0.5 mm thickness at cervical third and incisal two thirds respectively and length equal to that of the respective clinical crown. The veneers were then glazed in a ceramic oven.

### **III. EXPERIMENTAL DESIGN:**

Total of 40 teeth samples were taken for the study comprising of maxillary central incisors (Fig 2). Total of 40 teeth samples were randomly divided into four groups. Each group comprises 10 teeth. The four groups are of the following,

- 1) Group A:** No pre-surface treatment (control group)
- 2) Group B:** Ceramic veneers were treated with 1.23% APF gel for 10 minutes (Fig 3). Then the specimens were ultrasonically cleaned for 3 minutes in distilled water.
- 3) Group C:** Ceramic veneers sandblasted with 50 µm aluminium oxide at 60 psi at 0.5 Mpa for 5 seconds at a distance of 10 mm (Fig 5), then ultrasonically cleaned for 3 minutes in distilled water.
- 4) Group D:** Ceramic veneers were acid etched with hydrofluoric acid of 5% for 20 seconds (Fig 4). Then the specimens were ultrasonically cleaned for 3 minutes in distilled water.

### **IV. CEMENTATION OF VENEERS TO THE TOOTH SAMPLES:**

#### **Cementation of laminates with Variolink- N: (Fig 6)**

Before the cementation procedures, the tooth samples were cleaned with pumice flour with the polishing cup with the help of contra angle hand piece (NSK) in order to remove debris, smear layer (Fig -7). The 40 prepared teeth were acid etched for 15 seconds with 35% phosphoric acid gel and thoroughly rinsed with water for 30 seconds. The Excite DSC total etch adhesive was applied on the tooth surface and

Monobond-S, silane coupling agent was applied on to the laminates. Then both were cured according to the manufactures instructions. Cement was a combination of 25% Variolink yellow base, 25% Variolink white base, and 50% catalyst. The base and catalyst were then dispensed with auotmixing pad in the ratio of 1:1 ratio. Then it is mixed for 10 seconds and then applied on to the surface treated laminates and finally placed on the tooth surface (Fig 8).

The surface treated ceramic veneers were seated on the prepared teeth with light finger pressure and excess cement was removed with an explorer before an oxygen blocking gel (glycerin gel) was applied to the margin and the cement was further polymerized using a light cure unit (HIFLEX) (Fig 9 and 10) for 40 seconds. Margins were then finished with finishing diamond burs. The surface of the test samples was calculated using electronic Vernier caliper (DIGIMATIC) (Fig 11). In this manner, forty samples were cemented to the enamel with three different surface pre-treatment of porcelain laminates.

## **V. MEASUREMENT OF SHEAR BOND FAILURE LOADING BY UNIVERSAL TESTING MACHINE:**

The luted teeth were stored in distilled water at 37<sup>0</sup> C for 24 hours. AUTOMATIC Universal testing machine (AG-IS, SAHIMADZU) (Fig 12) .The force was applied at 90<sup>0</sup> to the long axis of the tooth. The acrylic mold was mounted in the lower member (Fig 13) and the upper member had the mono-bevel chisel with a cross head. A shear force was applied to the ceramic test sample at a cross head speed of 1.0mm / min until fracture occurred (Fig 14 and 15) and maximum load recorded for each specimen. The recorded values were then divided by the surface area of the sample to obtain the shear bond strength values in MPa. A total of 40 test samples

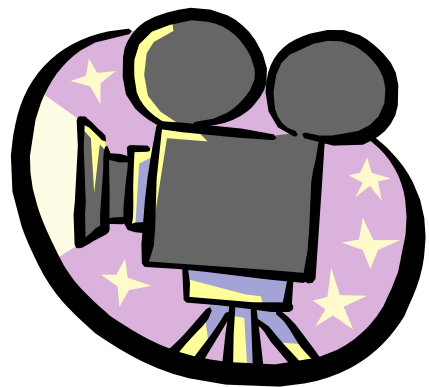
were tested in identical manner (Fig 16) and the shear bond strengths were tabulated for statistical analysis.

## **VI. STATISTICAL EVALUATION:**

All the statistical tabulations were done using Microsoft Excel (Microsoft, U.S.A.). The SPSS (SPSS for Windows 10.05, SPSS Software Corporation, Munich, Germany) software package was used for statistical analysis. One-way ANOVA was used to compare the mean values of the four groups (A, B, C and D). Tukey-HSD was used as the post hoc test and a p value < 0.05 was considered statistically highly significant.

## **VII. QUALITATIVE ANALYSIS OF SURFACE TOPOGRAPHY OF SURFACE TREATED, PREPARED TEETH SAMPLES USING SCANNING ELECTRON MICROSCOPE:**

SEM analysis was carried to identify surface topography and mode of failure, on one representative tested sample from each test group (Group A, Group B, Group C and Group D) after deboning of ceramic blocks, using a scanning electron microscope (HITACHI, S-3400N). The samples were placed on stubs, secured in place with an adhesive tape and coated with a thin layer of gold in a ion sputtering system (HITACHI,E-1010) (Fig 17). Coated samples (Fig 18) were loaded in scanning electron microscope (Fig 19) and examined under SEM to examine the surface topography of the samples 1000x magnifications (Fig 20).



***Photographs***

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Fig 1:- ARMAMENTARIUM

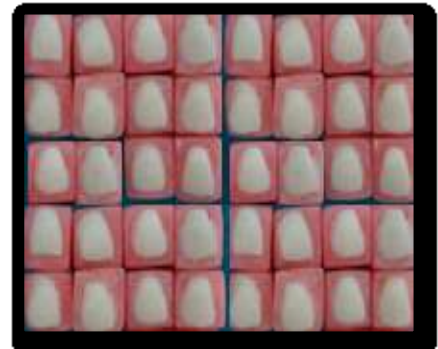


Fig 2:- TEETH SAMPLES  
EMBEDDED IN ACRYLIC



Fig 3:- APF GEL



Fig 4:- CERAMIC ETCHANT



Fig 5:- SANDBLASTING UNIT



Fig 6:- VARIOLINK CEMENT



Fig 7:- PREPARATION OF TOOTH SURFACE BEFORE LUTING



Fig 8:- RESIN CEMENTATION DONE ON THE SAMPLE





Fig 9:- LIGHT CURING UNIT



Fig 10:- CURING OF LAMINATES



Fig 11:- VERNIER CALIPER

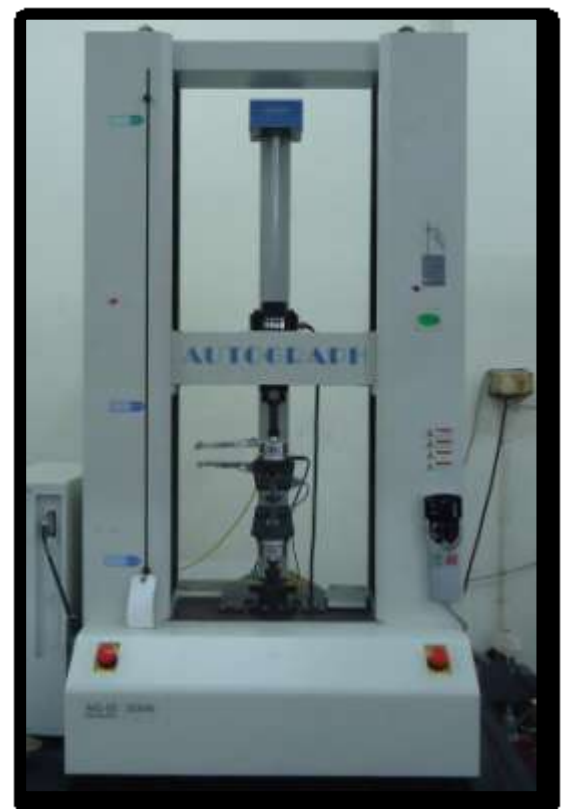


Fig 12:- UNIVERSAL TESTING MACHINE



Fig 13:- SHEAR LOADING OF  
SAMPLE



Fig 14:- TESTING INITIATED



Fig 15:- FRACTURE OF SAMPLES  
UNDER LOAD



Fig 16:- FRACTURED SAMPLES  
AFTER TEST



Fig 17:- ION SPUTTER



Fig 18:-SAMPLES AFTER  
GOLD SPUTTERING



Fig 19:- LOADING OF SAMPLES IN  
SCANNING ELECTRON MICROSCOPE



Fig 20:- SEM ANALYSIS



## *Results*

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The present in-vitro study was conducted to comparatively evaluate the shear bond strength between the intaglio surfaces of ceramic laminates subjected with three different types of surface pre-treatment, resin cement and the human tooth.

Forty recently extracted central incisors were sectioned and mounted in acrylic using a custom-made mounting jig. Porcelain laminates were fabricated on the teeth and were divided into four different groups as Group A: No surface pre-treatment, Group B: Etching with 1.23% APF gel for 10 minutes, Group C: Sandblasted with 50  $\mu\text{m}$  Aluminium oxide at 60 psi at 0.5 Mpa for 5 seconds at a distance of 10 mm and Group D: etching with 5%, Hydrofluoric acid for 20 seconds. All the groups except Group A were subjected to silanization after surface pre-treatment. All the porcelain laminates were bonded to the teeth with adhesive resin luting cement. Then, the samples were subjected to shear strength by Universal Testing Machine. The fracture load was noted in Newton and accordingly Shear bond strength was calculated in MPa. The results were tabulated and subjected to statistical analysis. One deboned test sample from each test group was randomly selected for a qualitative assessment by SEM analysis for surface topography. The results were tabulated and subjected to statistical analysis.

## **STATISTICAL ANALYSIS**

The mean value of the shear bond strength (MPa) of the four groups was given in table 1. The data was subjected to,

1. One way ANOVA analysis.
2. Tukey multiple comparison tests.

### **1. One – way ANOVA Test:**

This is employed to compare the means of three or more independent groups of observation. In One – way ANOVA test, the observed variability in the sample is subdivided into two components. Variability of the observations within a group about the group mean and variability of group means between groups about the overall mean. In this study, one way ANOVA was used to determine the statistical difference in shear bond strength within 4 groups.

### **2. Tukey – HSD (Honestly Significant difference) Test:**

A significant F and M statistic One – way ANOVA only indicates that the population means are probably unequal. It does not pin point where the differences are, so in order to determine this, multiple comparison tests are done. In this study, since significant differences were determined using One way ANOVA, the results were further analyzed using the Tukey – HSD test at a significant level of 5 %. This was done to determine where the differences between groups and within each group lie. All statistical analyses were calculated using the windows statistical software program. The results of one way ANOVA and Tukey HSD test were given in table.

**P value** 0.010-- significant at the level of 1%.\*\*

0.011- 0.050— significant at the level of 5%.\*

> 0.050--- non significant

**Table I** shows basic values and mean value of shear bond strength for Group A, Group B, Group C and Group D test samples: Shear bond strength in MPa

Sample No. (n)	Group A	Group B	Group C	Group D
<b>1</b>	10.8	10.45	20.58	21.5
<b>2</b>	10.4	14.15	13.67	23.67
<b>3</b>	10.3	11.67	16.07	20.07
<b>4</b>	9.9	13.24	11.43	24.43
<b>5</b>	8.50	12.23	12.78	22.78
<b>6</b>	11.25	15.40	14.97	22.97
<b>7</b>	13.14	14.96	11.45	19.45
<b>8</b>	9.15	12.14	19.87	17.87
<b>9</b>	10.26	11.78	20.56	25.56
<b>10</b>	12.36	14.23	15.21	26.21
<b>MEAN</b>	<b>10.60 ± 1.384</b>	<b>13.025 ± 1.618</b>	<b>15.659 ± 3.569</b>	<b>22.451 ± 2.710</b>

**Table II:** Comparison between mean shear bond strength values of Group A (control), Group B (APF gel), Group C (sandblasting) and Group D (HF acid) test samples using One-way ANOVA

GROUP	Number of Samples	Mean Shear Bond Strength (MPa)	Standard Deviation	P-value
<b>A</b>	10	10.60	<b>1.384</b>	<b>&lt;0.001</b>  <b>HS</b>
<b>B</b>	10	13.025	<b>1.618</b>	
<b>C</b>	10	15.659	<b>3.569</b>	
<b>D</b>	10	22.451	<b>2.710</b>	

P-value<0.05 denotes significance at the 5% level, HS – Highly Significant

**Table III:** Shows the comparison between mean shear bond strength values of Group A (control) and Group B (APF gel) test samples using Tukey HSD.

<b>GROUP</b>	<b>Number of samples</b>	<b>Mean Shear Bond Strength (MPa)</b>	<b>Standard Deviation</b>	<b>P-value</b>
<b>A</b>	10	10.60	<b>1.384</b>	<b>0.148</b>  <b>NS</b>
<b>B</b>	10	13.035	<b>1.618</b>	

p<0.05, NS – Non Significant

P-value<0.05 denotes significance at the 5% level

**Table IV:** Shows the comparison between mean shear bond strength values of Group A (control) and Group C (sandblasting) test samples using Tukey HSD.

<b>GROUP</b>	<b>Number of Samples</b>	<b>Mean Shear Bond Strength (MPa)</b>	<b>Standard Deviation</b>	<b>P-value</b>
<b>A</b>	10	10.60	<b>1.384</b>	<b>0.000</b>  <b>HS</b>
<b>C</b>	10	15.659	<b>3.569</b>	

p<0.05, HS – Highly Significant

P-value<0.05 denotes significance at the 5% level

**Table V** shows the comparison between mean shear bond strength values of Group A (control) and Group D (HF acid) test samples using Tukey HSD.

<b>GROUP</b>	<b>Number of samples</b>	<b>Mean Shear Bond Strength (MPa)</b>	<b>Standard Deviation</b>	<b>P-value</b>
<b>A</b>	10	10.60	<b>1.384</b>	<b>0.0000</b>  <b>HS</b>
<b>D</b>	10	22.451	<b>2.710</b>	

p<0.05, HS – Highly Significant

P-value<0.05 denotes significance at the 5% level



**Table VI** shows the comparison between mean shear bond strength values of Group B (APF gel) and Group C (sandblasting) test samples using Tukey HSD.

<b>GROUP</b>	<b>Number of Samples</b>	<b>Mean Shear Bond Strength (MPa)</b>	<b>Standard Deviation</b>	<b>P-value</b>
<b>B</b>	10	13.025	<b>1.618</b>	<b>0.101</b>  <b>NS</b>
<b>C</b>	10	15.659	<b>3.569</b>	

p<0.05, NS – Non Significant

P-value<0.05 denotes significance at the 5% level

**Table VII** shows the comparison between mean shear bond strength values of Group B (APF gel) and Group D (HF acid) test samples using Tukey HSD.

<b>GROUP</b>	<b>Number of Samples</b>	<b>Mean Shear Bond Strength (MPa)</b>	<b>Standard Deviation</b>	<b>P-value</b>
<b>B</b>	10	13.025	<b>1.618</b>	<b>0.000</b>  <b>HS</b>
<b>D</b>	10	22.451	<b>2.710</b>	

p<0.05, HS – Highly Significant

P-value<0.05 denotes significance at the 5% level

**Table VIII** shows the comparison between mean shear bond strength values of Group C (sandblasting) and Group D (HF acid) test samples using Tukey HSD.

<b>GROUP</b>	<b>Number of Samples</b>	<b>Mean Shear Bond Strength (MPa)</b>	<b>Standard Deviation</b>	<b>P-value</b>
<b>C</b>	10	15.659	<b>3.569</b>	<b>0.000</b>  <b>HS</b>
<b>D</b>	10	22.451	<b>2.710</b>	

p<0.05, HS – Highly Significant

P-value<0.05 denotes significance at the 5% level

Descriptives				
VAR00002				
	N	Mean	Std. Deviation	Std. Error
GROUP A	10	10.6060	1.38482	.43792
GROUP B	10	13.0250	1.61866	.51187
GROUP C	10	15.6590	3.56929	1.12871
GROUP D	10	22.4510	2.71091	.85726
Total	40	15.4353	5.07800	.80290

ANOVA					
VAR00002					
	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	784.018	3	261.339	42.448	.000
Within Groups	221.640	36	6.157		
Total	1005.658	39			

Multiple Comparisons				
VAR00002			Tukey HSD	
GROUPS	Compared with	Mean Difference (I-J)	Std. Error	Sig.
A	B	2.41900	1.10965	.148
	C	5.05300*	1.10965	.000
	D	11.84500*	1.10965	.000
B	A	2.41900	1.10965	.148
	C	-2.63400	1.10965	.101
	D	-9.42600*	1.10965	.000
C	A	5.05300*	1.10965	.000
	B	2.63400	1.10965	.101
	D	-6.79200*	1.10965	.000
D	A	11.84500*	1.10965	.000
	B	9.42600*	1.10965	.000
	C	6.79200*	1.10965	.000
*. The mean difference is significant at the 0.05 level.				

The statistical analysis shows the following-

**Table II:** Shows the comparison between mean shear bond strength values of Group A (control), Group B (APF gel), Group C (sandblasting) and Group D (HF acid) test samples using One-way ANOVA  $10.60 \pm 1.384$ ,  $13.025 \pm 1.618$ ,  $15.659 \pm 3.569$ ,  $22.451 \pm 2.710$  respectively.

**Table III:** Shows the comparison between mean shear bond strength values of Group A (control) and Group B (APF gel) test samples using Tukey HSD 0.148,  $p < 0.05$ , NS – Non Significant

P-value  $< 0.05$  denotes significance at the 5% level

**Table IV:** Shows the comparison between mean shear bond strength values of Group A (control) and Group C (sandblasting) test samples using Tukey HSD 0.000,  $p < 0.05$ , HS-Highly Significant

P-value  $< 0.05$  denotes significance at the 5% level

**Table V:** Shows the comparison between mean shear bond strength values of Group A (control) and Group D (HF acid) test samples using Tukey HSD 0.00,  $p < 0.05$ , HS – Highly Significant.

P-value  $< 0.05$  denotes significance at the 5% level

**Table VI:** Shows the comparison between mean shear bond strength values of Group B (APF gel) and Group C (sandblasting) test samples using Tukey HSD 0.101  $p < 0.05$ , NS – Non Significant.

**Table VII:** Shows the comparison between mean shear bond strength values of Group B (APF gel) and Group D (HF acid) test samples using Tukey HSD 0.000,  $p < 0.05$ , HS – Highly Significant.

**Table VIII:** Shows the comparison between mean shear bond strength values of Group C (sandblasting) and Group D (HF acid) test samples using Tukey HSD 0.00,  $p < 0.05$ , HS – Highly Significant.

**Interpretation of results:**

On comparison between the mean shear bond strengths of Group A, Group B, Group C and Group D in (Table II), using One-way ANOVA it was found that there was a statistically significant difference between the mean shear bond strength of the four groups. Group D (HF etching) had the highest mean shear bond strength followed by Group C (sandblasting) and the lowest shear bond strength value was observed in Group A (control).

On comparison between the mean shear bond strengths of Group A and Group B (Table III) it was found that Group B had exhibited a higher mean shear bond strength value compared to Group A. On statistical analysis using Tukey HSD, it was found that the p-value  $> 0.05$ , denoting no statistically significant difference between these two groups

On comparison between the mean shear bond strengths of Group A and Group C (Table IV) it was found that Group C had exhibited a higher mean shear bond strength value compared to Group A. On statistical analysis using Tukey HSD, it was found that the p-value  $< 0.05$ , denoting highly significant difference between these two groups.

On comparison between the mean shear bond strengths of Group A and Group D it was found that Group D (Table V) had exhibited a higher mean shear bond strength value compared to Group A. On statistical analysis using Tukey HSD, it was

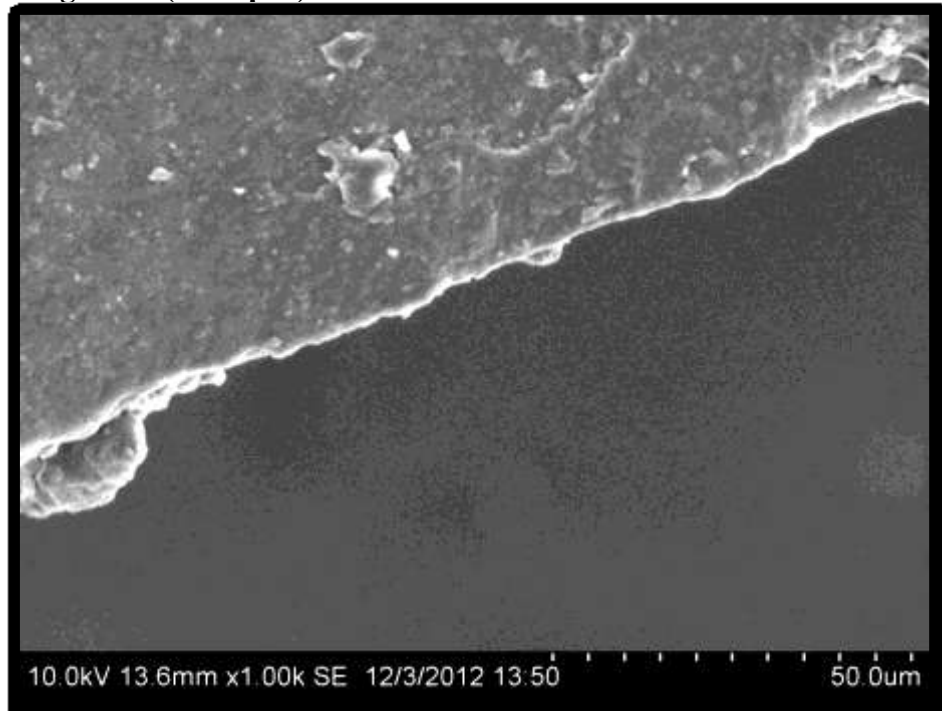
found that the p-value  $<0.05$ , denoting a statistically significant difference between these two groups.

On comparison between the mean shear bond strengths of Group B and Group C it was found that Group C (Table VI) had exhibited a higher mean shear bond strength value compared to Group B. On statistical analysis using Tukey HSD, it was found that the p-value  $>0.05$ , denoting no statistically significant difference between these two groups.

On comparison between the mean shear bond strengths of Group B and Group D it was found that Group D (Table VII) had exhibited a higher mean shear bond strength value compared to Group B. On statistical analysis using Tukey HSD, it was found that the p-value  $<0.05$ , denoting a statistically highly significant difference between these two groups.

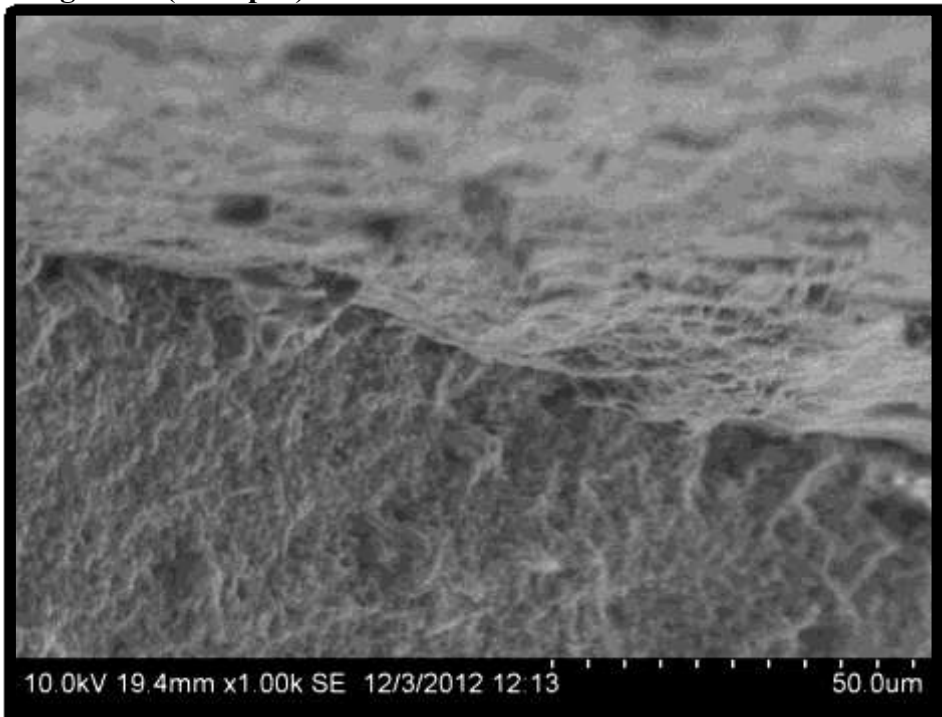
On comparison between the mean shear bond strengths of Group C and Group D it was found that Group D (Table VIII) had exhibited a higher mean shear bond strength value compared to Group C. On statistical analysis using Tukey HSD, it was found that the p-value  $<0.05$ , denoting a statistically highly significant difference between these two groups.

**1) SEM figure 1: (Group A)**



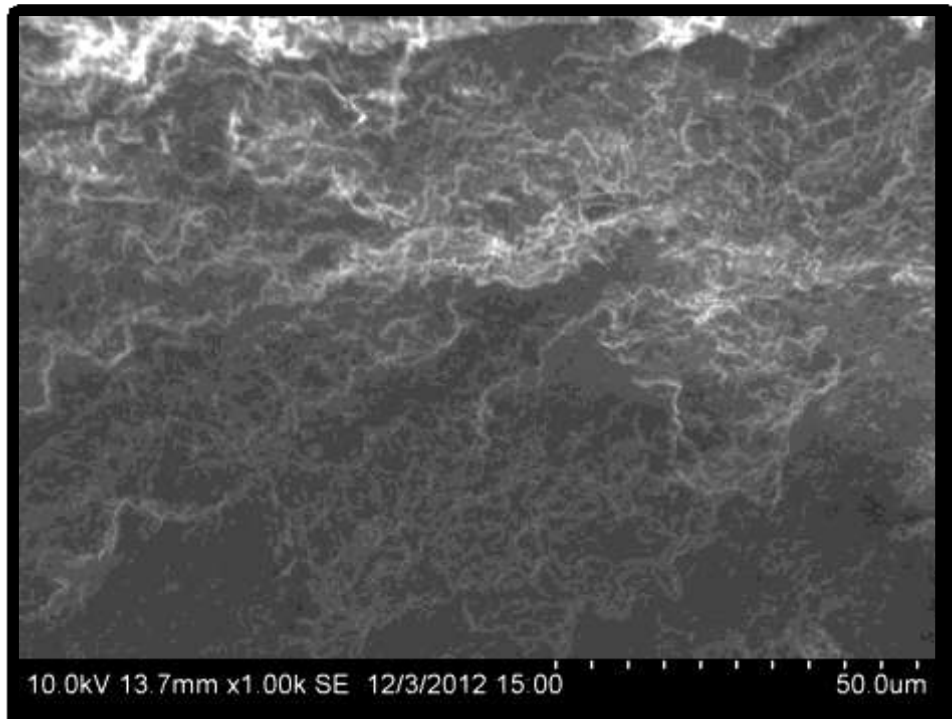
Original magnification X 1,000

**2) SEM figure 2: (Group B)**



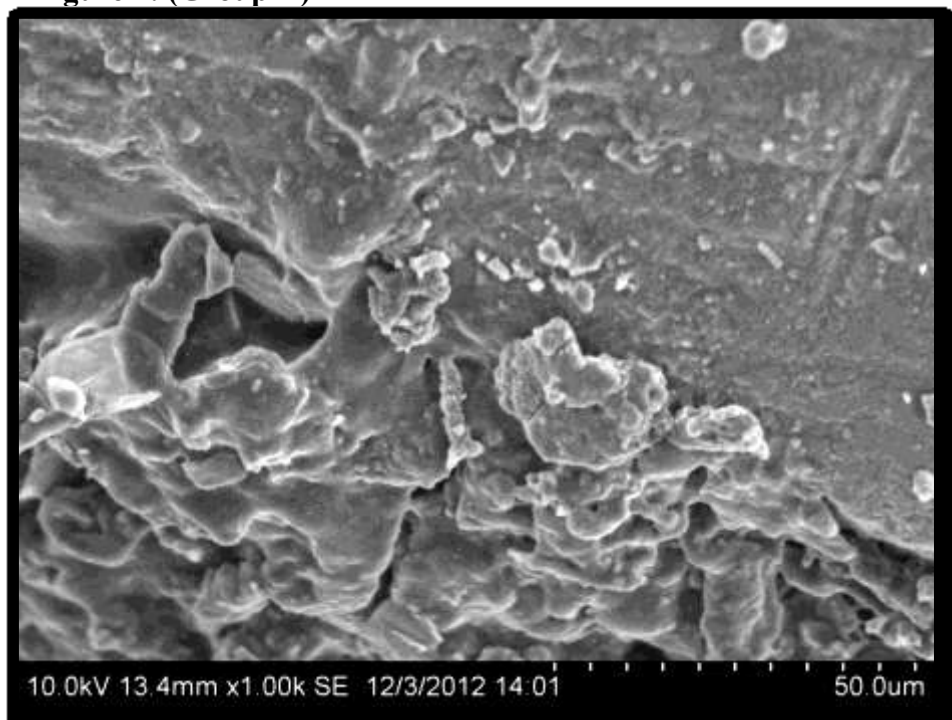
Original magnification X 1,000

**3) SEM figure 3: (Group C)**



Original magnification X 1,000

**4) SEM figure 4: (Group D)**



Original magnification X 1,000

**Qualitative analysis of surface topography of pre-treated laminate surfaces of Group A, Group B, Group C and Group D by scanning electron microscope under 1000x magnification.**

**SEM fig 1:-** The SEM photomicrograph of the Group A sample, with no pre-surface treatment shows a uniform, smooth, flat appearance of the adhesive junction at a 1000x magnification.

**SEM fig 2:-** The SEM photomicrograph of the Group B sample, surface pre-treated with 1.23% APF gel shows a slightly roughened surface with many irregularities and ill-defined micro porosities in some areas.

**SEM fig 3:-** The SEM photomicrograph of the Group C sample, surface pre-treated with 50 µm aluminium oxide shows an etched pattern with well defined small grains.

**SEM fig 4:-** The SEM photomicrograph of the Group D sample, surface pre-treated with 5% hydrofluoric acid shows an etched relief pattern with cotton wool appearance which presented as a porous and dendritic appearance, sufficient for creating micromechanical retention.





# *Discussion*

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“Biomimetics”, a newly emerging interdisciplinary material science refers to material processing in a manner similar to the oral cavity and mimicking or recovery of the biomechanics of the original tooth by the restorative material.<sup>21</sup>

It is based on the assumption that, the hardness of enamel protects the soft underlying dentin. On the other hand, the crack-arresting effect of dentin and of the thick collagen fibres at the dentinoenamel junction compensate for the inherently brittle nature of enamel. This structural and physical interrelationship between an extremely hard tissue and a more pliable, softer tissue provides the natural tooth with its unique ability to withstand masticatory and thermal loads during a lifetime. Similarly, it is expected that the biomechanical and structural integrity of the enamel-dentin complex could be partially mimicked using porcelain veneers, because of the improvement of adhesive procedures. So the concept of porcelain veneers is based on the “Biomimetics” concept.<sup>21</sup>

The porcelain veneer technique includes the bonding of a thin porcelain laminate to the tooth surface using adhesive techniques. The success of the porcelain veneer is greatly determined by the strength and durability of the formed bond between the three different components of the bonded veneer complex, as there are the tooth surface, the luting agent and the porcelain veneer.<sup>23</sup>

IPS Empress 2 a pressable multiphase popular glass ceramic with a high degree of crystallinity was developed in 1998 with the same laboratory procedure and equipment used for the initial version of IPS Empress. IPS Empress 2 is composed of approximately 70 vol% leucite, which confers improved mechanical properties. This material has generated considerable interest because of its ease of fabrication based on the lost wax technique and excellent esthetic feature. So this material was used in our study and veneering material consisted of an apatite glass ceramic.<sup>10, 31</sup>

Adhesive and restorative success for any indirect restoration begins and ends at the restorative-tooth interface. The bonded restorative complex includes the outer layers of the substrate, the adhesive layer, and the restorative material. Any restoration when properly joined to the tooth substrate is able to provide an improved marginal seal while reducing marginal contraction gaps, microleakage, nanoleakage, marginal staining, secondary caries and biologically preserves tissues providing long-term functional success.<sup>30</sup>

So the cementation process is vital for the clinical success of all-ceramic restorations. The success of the cementation process is dependent on the composition of the ceramic material. When zinc phosphate or glass ionomer cements are used, mechanical retention is necessary. Such water based cements work mainly by frictional force. On the other hand, when mechanical retention is compromised, adhesive luting systems are recommended.<sup>32</sup>

Adhesive bonding is dependent on the surface energy and wettability of the adherend by the adhesive. Porcelain laminate veneers and the crowns rely on the synergistic bonding achieved between the porcelain and resin cement in order to survive the rigours of the oral environment.<sup>13</sup> Bonding to lithium disilicate based ceramics is usually obtained by two simultaneous mechanisms, namely<sup>10</sup>

- a) Micromechanical retention provided by preconditioning of the ceramic surface

- b) Chemical coupling by the application of a silane coupling agent

The bond of the resin luting cement to the tooth structure is enhanced by acid etching of enamel or dentin and by the use of a dentin adhesive. The penetration of monomers into the demineralized dentinal matrix, followed by polymerization, promotes the micromechanical bond via hybrid layer formation. The same principle of

this retention process can be similarly reproduced in the intaglio surface of ceramic or laboratory-processed composite resin restorations through the use of different preconditioning treatments.<sup>32,37</sup>

The aim of pre-cementation surface modification of the porcelain is to modify its structure, to increase the surface area available for bonding and to create undercuts that increase the micromechanical retention and strength of the bond to the resin luting cement.<sup>9,13</sup>

It is not clear whether mechanical roughening (with the use of air particle abrasion, sandblasting, grinding by diamond burs, or acid etching by hydrofluoric acid or ammonium bifluoride or acidulated phosphate fluoride gel), chemical bonding (with silane), or some combination of the two is the most effective surface treatment for bonding ceramic restorations with resin cements.<sup>7,9,27,29,32</sup>

So the clinicians are often confused regarding the most effective way to treat the intaglio surfaces of indirect porcelain restorations prior to placement with various adhesives and luting resins. They are often equally perplexed about the “ideal” surface treatment for the intraoral repair of pre-existing porcelain restorations.<sup>14,27</sup>

This study was taken up to clear the air about these pre-surface conditioning methods

- a) Without pre-surface conditioning was used as control,
- b) Acidulated phosphate fluoride gel with silane,
- c) Sandblasting with silane and
- d) Hydrofluoric acid with silane.

HF is an inorganic acid capable of etching glass surfaces. It has been used for hundreds of years to create decorative designs and patterns on glass and various ceramic materials.<sup>14</sup> Horn in 1983 proposed etching porcelain laminate veneer

restoration with either hydrofluoric acid or Stripit solution, and it is a standard protocol to bond etchable porcelains to teeth.<sup>8,13,28</sup>

As per the recommendations of the manufacturer of IPS Empress 2, we used < 5% HF with a very specific etching time of only 20 seconds.<sup>14</sup> Care was taken not to over etch, as it would affect significantly the bond strength.

Basic chemistry has shown that when an acid and base react, various salts are produced as by-products of the reaction. In the case of HF etching, the porcelain acts as a base, and when it reacts with HF, various insoluble metallic salts are formed. The white residue consists not merely of “porcelain salts,” but also of numerous microscopic crystalline fragments exposed, possibly weakened by HF exposure, and displaced after the glassy matrix supporting them has been dissolved by HF and the porcelain surface washed and dried.<sup>14</sup>

Hydrofluoric acid is known to be a hazardous substance, so we were very careful in handling and storage of this material. Considering the hazardous effect of hydrofluoric acid solution, it's even recommended to be applied as a gel material to prevent its volatilization or dripping.<sup>49</sup>

Utmost care was exercised during this pre-treatment, as contamination with latex gloves, saliva, fit-checker paste or even try-in-paste would lower the bond strength and etched restorations were ultrasonically cleaned in water for 20 minutes and dried.<sup>23</sup>

Silanes are a class of organic molecules that contain one or more silicon atoms. They are adhesion promoters, useful as a chemical coupler linking, organics (i.e., resin-based materials) to inorganics (e.g., porcelain, some oxidized metals, and glass fillers in resin-based composites).<sup>14,36</sup> Rochette first advocated the use of silane

as a coupling agent, and Lacy et al showed that silane treatment increased almost 5 times higher bond strength than that of acid etched porcelain surface.<sup>28</sup>

Etching and silanization increases the surface energy and the wettability of the ceramic substrate, which decreases, the contact angle between the ceramic surface and the resin cement.<sup>10</sup>

Resin luting cements are a mix of mono functional monomers with a variable amount of (55-70%, v/w) of filler of varying sizes, forms and composition. The amount of filler is reduced in comparison to restorative resins, in order to decrease viscosity and allow better adaptation of a rigid restoration to a cavity surface. Among resin cement's advantages are: colour, adhesion to dental tissues and other materials, reduced solubility, improved mechanical properties in comparison to traditional cements and the possibility of light or dual polymerization. However, there are several disadvantages, such as the need for careful manipulation, dry operating filed during bonding and polymerization contraction.<sup>6</sup> They are classified according to their initiation mode as autopolymerising (chemically activated), photoactivated, or dual activated materials.<sup>29</sup>

In this study, we had used dual-activated resin cements as they offer extended working times and controlled polymerization, although chemical activators ensure a high degree of polymerization.<sup>29</sup> The excess non-polymerised resin cement was removed with a brush moistened with bonding resin. This will reduce the dragging out tendency of the resin out of the marginal gap and ensure a smoother margin that is polishable.<sup>23</sup>

#### **Bond strength of pre-surface treatment with HF+Silane: -Table I and Graph I**

In this study, a total of 10 samples were etched with hydrofluoric acid and subsequently treated with silane. The mean bond strength was **22.451 ± 2.710 MPa**

with lowest and highest being **17.87 MPa** and **26.21 MPa** respectively. Our results were very much similar to that of Spohr et al<sup>31</sup> who got  $25.6 \pm 1.2$  MPa in groups treated with HF+S. The results were in accordance with the results of Stewart GP et al<sup>27</sup> who also got the results in the range of 16.0 to 21.7 MPa at 24 hours and 15.9 to 21.8 MPa after 6 months of storage in saline solution. The results were also in the range of the findings of Proenca et al<sup>10</sup> who got mean microtensile bond strength of 18.8 MPa, 17.4 MPa and 15.7 MPa in different groups of resin cements, but all treated with hydrofluoric acid and silane. Slightly lower values of mean microtensile bond strength were obtained by Vasconcellos WA et al<sup>43</sup> ( $16.05 \pm 4.32$ ) in groups treated with HF, where as much higher values were obtained by Bona AD et al<sup>38</sup> ( $31.9 \pm 8.6$  MPa), Filho AM et al<sup>36</sup> ( $56.8 \pm 10.4$  MPa), Nagai T et al<sup>33</sup> ( $46.6 \pm 5.2$  MPa).

The differences in the values of different investigators is mainly attributed to the differences in ceramic microstructure, concentration of HF, etching patterns, etching time, type of silane, method of silane application, thermocycling, storage time between preparation of the test specimen and the test itself, storage media before testing, and testing methods.

For example, most of the above mentioned investigators used IPS Empress 2 ceramic material, while Stewart GP et al<sup>27</sup> used Ceramco II. Spohr et al<sup>31</sup> used 10% HF concentration applied for one minute, where as in this study, we had used <5% HF applied for 20 seconds. In the studies like by Vasconcellos WA et al<sup>43</sup>, only HF was used without silane which might have contributed to the lower bond strength.

The type of silane used also influences the bond strength. In this study, we used Monobond-S ceramic primer, a single-bottle silane, which was pre-hydrolyzed by the manufacturer consisted of 5% silane in a water/ethanol solution with an acetic acid adjusted pH of 4 to 5. We stored the silane in a refrigerator since once it is

hydrolyzed, silane molecules have a tendency to react with one another, forming high-molecular weight oligomers (i.e. polysiloxanes) that can actually function as a lubricant and potentially decrease bond strength to porcelain. The refrigerated silane was brought to room temperature prior to use in this study. Only a single coat of silane was applied then allowed to evaporate for 3 minutes and air-dried for 30 seconds.

Excessive application of silane will create an unnecessarily too thick and intrinsically weak layer, which could be prone to cohesive failure. Clinically, the surface of the porcelain should not look shiny after silane application and drying. A shiny surface on the porcelain after silane application and drying could be an indication of excessive silane deposition and, if seen, the surface should be sandblasted under low pressure, re-etched with HF, cleaned with ethanol in an ultrasonic, and the silane re-applied. A properly silane-treated porcelain veneer visually appears essentially the same as it did prior to placing the silane (i.e., matt/dull finish).<sup>14</sup> Also, the silane application time, evaporation time, air-drying time varied from study to study.

Barghi, Berry, and Chung demonstrated that different silanes yield different bond strength values. These authors also found differences in bond strengths following thermal treatment of silanized porcelain using two-bottle silanes. Different silanes may present in their composition different solvents which will influence their reactivity and stability in various ways.<sup>36</sup> All these factors might had an influence on the final bond strength.

The ceramic-composite bond is susceptible to chemical, thermal, and mechanical influences under intraoral conditions. The simulation of such influences in the laboratory is compulsory to draw conclusions on the long-term durability of a



specific bonding procedure and to identify superior materials and techniques. Long-term water storage and thermocycling of bonded specimens are accepted methods to simulate aging and to stress the bonding interface.<sup>17,29</sup>

In this study, thirty minutes after bonding, the samples were immersed in 37<sup>0</sup> C water for 24 hours. The storage time of 24 hours was chosen in this study because it was convenient and, theoretically, it was long enough to allow the complete polymerization of resin cement. Also, it is a reasonable period when the clinician proceeds with the occlusal adjustment and removes excess cement, and, therefore, may stress the bond.<sup>22</sup> The above mentioned studies used different storage timings, different storage media and some of the studies even performed thermocycling procedures. Thermocycling of the samples, which was not done in this study, might have had a significant effect on the results.

The differences in the mean bonding area of different studies had a very significant effect on the bond strength. The shear bond strength is calculated by the force to break divided by bonding surface area. If the bonding area were smaller, then the bonding strength value would be higher. Also, the crosshead speed of the universal testing machine have a impact on shear bond strength and it was concluded that the slower the crosshead speed, the lower the shear bond strength.<sup>28</sup> This explains the differences in the values of final bond strength.

Proenca et al,<sup>10</sup> Vasconcellos WA et al,<sup>43</sup> Bona AD et al,<sup>38</sup> and Filho AM et al<sup>36</sup> used mean microtensile bond strength methods, where as in this study, we adopted the most common shear bond strength. There were various types of mechanical bond strength tests. Some of these authors stated that, as demonstrated by finite element stress analyses, the non uniformity of the interfacial stress distribution generated during conventional tensile and shear bond strength testing may result in

fracture initiation from flaws at the interface or within the substrate in regions of high localized stress. This manner of fracturing provides only limited information about the true bond strength.<sup>10,36</sup>

Airborne particle abrasion with aluminium trioxide particles which has been introduced in the late 1980s has been proven to be effective for conditioning ceramics. The blasting pressure causes these alumina particles to be embedded within the surface. Following the application of a silane coupling agent, the modified surface structure is thereby rendered more reactive with the resin, hence enabling chemical adhesion between both surfaces.<sup>41</sup>

#### **Bond strength of presurface treatment with Sandblasting + Silane: Table I and Graph I**

In this study, a total of 10 samples were sandblasted with 50 µm aluminium oxide at 60 psi at 0.5 Mpa for 5 seconds at a distance of 10 mm, then ultrasonically cleaned for 3 minutes in distilled water and subsequently treated with silane. The mean bond strength was **15.659 ± 3.569 MPa** with lowest and highest being **11.43 MPa and 20.58 MPa** respectively.

In a study by Kansu G et al<sup>53</sup> a shear bond strength of 11.94 ± 2.94 MPa was obtained when IPS Empress 2 ceramic was surface pre-treated with sandblasting with 50 µm Aluminium oxide particles, the results of which were slightly similar to our study. Our results were very much similar to that of Vasconcellos WA et al,<sup>43</sup> who also got 14.03±5.52 MPa with sandblasting of IPS Empress 2 ceramic material, but without silane treatment. Slightly lower values like 11.8 ± 1.0 MPa were obtained by Spohr et al.<sup>31</sup>

These differences could be explained on the basis of differences in the choice of selection of mechanical tests. Slightly higher values were obtained by Menezes

FCH<sup>52</sup> who got values of around  $23.37 \pm 3.85$  MPa with sandblasting of IPS Empress 2 ceramic material. Similar values like  $14.4 \pm 4.6$  MPa,  $18.0 \pm 4.1$  MPa were also observed in a study by Begazo CC et al<sup>34</sup> with different types of resin cements luted on Synthoceram, an aluminium oxide-reinforced glass ceramic core material.

Slightly similar values of bond strength of around  $19.2 \pm 5.1$  MPa,  $23.1 \pm 4.8$  MPa,  $9.5 \pm 3.0$  MPa,  $8.4 \pm 3.6$  MPa were observed in a study by Stewart GP et al<sup>27</sup> when Ceramco II was treated with different types of resin cements. Madani M et al<sup>26</sup> has got  $22.35 \pm 5.98$  MPa with sandblasted In-Ceram ceramic, Nagayassu MP et al<sup>9</sup> has got values of  $10.4 \pm 2.37$  MPa with only sandblasting without silane treatment of 50% aluminous porcelains, Lee JY et al<sup>8</sup> has got the values of around  $20.88 \pm 2.75$  MPa with sandblasting and silane treatment of Authentic porcelain, Özcan M et al<sup>7</sup> had observed bond strength of  $12.9 \pm 2.0$  MPa,  $10.2 \pm 0.7$  MPa with different types of resin cements treated with In-Ceram porcelains.

The comparative difference between the bond strength of this study with other studies and this is mainly attributed to the types of porcelain microstructure. In this study, we had used lithium disilicate reinforced porcelain, where as in most of the studies, the type of porcelain was different. This contributed to the main difference in the final result.

Through this study, we also noted that there are very few studies conducted on lithium disilicate ceramics upon sandblasting type of surface pre-treatment. The other attributes to the difference in the bond strength are size of the aluminium oxide particles, bar pressure at which sandblasting was done, distance at which pressure was applied, time of application of pressure and even the surface contamination after sandblasting procedures. Even contamination with hands, gloves or saliva tends to decrease the bond strength. Usually it is advocated that after sandblasting the test

specimens must be ultrasonically cleaned. It is not mentioned in some of the studies. So all these factors will lead to differences in the final results of bond strength between various studies.

Sandblasting of ceramic restorations has the potential to remove significant amounts of material and could affect the clinical fit of the restoration. Sandblasting of the feldspathic-based Empress ceramic yielded a detrimental surface volume loss of almost 1 mm<sup>3</sup> which is almost 36 times greater than the loss for In-Ceram ceramic. The dramatic abrasion and volume loss pattern for Empress ceramic under these conditions was clinically significant. Empress ceramic sustained a great deal of damage during the sandblasting, and it is assumed that other feldspathic ceramics would behave similarly.<sup>16</sup>

#### **Bond strength of control group: - Table I and Graph I**

In this study, a total of 10 samples were not subjected to any pre-surface treatment and were used as controls. The mean bond strength of this control was **10.60 ± 1.384 MPa** with lowest and highest being 8.50 MPa and 13.14 MPa respectively. Our findings are in correlation with the findings of Proenca JP et al,<sup>10</sup> who also got the somewhat similar results like 9.6 ± 1.9 MPa, 6.2 ± 1.2 and 7.4 ± 1.9 MPa with three different types of resin cements

#### **Bond strength of pre-surface treatment with APF gel + Silane: - Table I and Graph I:**

In this study, a total of 10 samples were etched with 1.23% acidulated phosphate gel for 10 minutes. Then the specimens were ultrasonically cleaned for 3 minutes in distilled water and subsequently treated with silane. The mean bond strength was **13.025 ± 1.618 MPa** with lowest and highest being **10.45 MPa and 15.40 MPa** respectively.

APF gel, widely used for in-office fluoride application, consists of sodium fluoride, phosphoric acid, and hydrofluoric acid. It is safe for oral tissue, unlike hydrofluoric acid, which can produce tissue rash and burn. Consequently, APF gel has been proposed as an alternative for ceramic surface etching before bonding with composite resin.<sup>54</sup>

Kewalin et al, showed that a seven to 10-minute application of 1.23% APF gel on a leucite containing porcelain produced a shear bond strength to composite similar to a four-minute etch with 9.6% HF.<sup>54</sup> Other studies have also shown that etching with APF, even with prolonged application times, results in very shallow etching patterns when compared to HF etching for much shorter time periods.<sup>14</sup> In addition, 10-minute etching with APF gel is relatively time consuming compared to hydrofluoric acid etching.

#### **Comparison of all groups (Group A, Group B, Group C and Group D) using One-way ANOVA Table II and Graph II:**

Among all the four groups, it was noted that the Group D (HF + Silane) produced the highest bond strength of **22.451 ± 2.710 MPa** which is statistically highly significant with p-value <0.001.

Hydrofluoric acid in combination with silane group was the highest among the four different types of surface treatments. The possible explanation could be as follows, IPS Empress 2 glass ceramic is formed by elongated crystals of lithium disilicate. A second phase is composed of lithium orthophosphate. A glass matrix surrounds both crystalline phases. Hydrofluoric acid attacks the glass phase of ceramics, partially dissolving it to the depth of a few microns and, as a result, the lithium disilicate crystals protrude from the glassy matrix. This treatment significantly

changes the surface morphology, increasing the surface area and irregularities within the lithium disilicate ceramic. This will favour the infiltration and retention of adhesive materials and made the ceramic surface more retentive.<sup>10,14,29,31,32,36</sup>

Some studies indicated that acid etching of ceramic could be eliminated, resulting in a reduction in operating time and elimination of the hazard of storing hydrofluoric acid. Only the application of silane would give bond strengths comparable with acid etching and surpass the ceramic's own cohesive strength.<sup>31</sup>

This may not be necessary for the IPS Empress 2 ceramic framework, since the ceramic retentive surface promoted by acid etching was sufficient to obtain the highest bond strengths. Another factor to be considered is the higher cohesive strength of the IPS Empress 2 ceramic framework in comparison to feldspathic ceramics.<sup>31</sup>

#### **QUALITATIVE ANALYSIS OF SURFACE TOPOGRAPHY OF SURFACE TREATED, PREPARED TEETH SAMPLES USING SCANNING ELECTRON MICROSCOPE.**

Specimens were sputter-coated with gold and examined under Scanning Electronic Microscope at 10 KV. Areas that represented the average roughness or topographical contours of each ceramic specimen were viewed and photographed at original magnification X1000.

A very flat surface without any kind of irregularities was noted in SEM fig 1 (Group A). So there was a remarkable reduction in the bond strength in this group which was well evidenced in the shear bond results. The adhesive type of failure was observed in this group.

APF gel etchant seemed to build up surface deposits preferentially on the lithium disilicate crystals which were seen in SEM fig 2 (Group B). The etching with

APF gel resulted only in the slight roughened surface with very little increase in the surface area which might not played a vital role in the adhesive process and showed cohesive mode of failure.

An irregular relief pattern with loss of surface structure was generated after air-borne particle abrasion with alumina was noted in SEM fig 3 (Group C). Alumina abrasion considerably roughened the Empress 2 surface. However, reduction in bond strength was remarkable. A particle-abraded surface was not mechanically retentive, although the surface appeared to be more retentive than polished or glazed surfaces and mixed type of failure was seen. The findings were in supportive of our shear bond strength results of Group C. Similar opinion were also coated by various authors.<sup>8,31,33</sup>

In the present study, etching with hydrofluoric acid produced remarkable morphological alterations on ceramic surface, which presented a porous and dendritic or in some areas honeycomb like appearance, sufficient for creating micromechanical retention SEM fig 4 (Group D). Hydrofluoric acid was able to remove the glass matrix and lithium orthophosphate crystalline phase, thus creating irregularities within the lithium disilicate crystals. The micrograph of the present study suggested that hydrofluoric acid attacked both lithium orthophosphate crystalline phase and glass matrix. This was confirmed by the fact that etched surface displays both attacked crystals and etched glass matrix. These findings were similar to that of other studies.<sup>7,9,23,31,33</sup> The findings of this study were also supported by the shear bond strength results. This group showed mixed mode of failure at the laminate, tooth and luting agent complex.

# *Summary and Conclusion*

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This study was done to Compare and Evaluate the **Shear bond strength** of four different pre-surface treatments of porcelain laminates to human dental hard tissue.

The number of samples for each group were ten. Ceramic Laminates were fabricated and pre-surface treatment was done and divided into Group A (no pre-surface treatment, acted as control group), Group B (APF gel + silanization), Group C (sandblasting + silanization) and Group D (HF + silanization). The teeth surfaces were etched with 37% phosphoric acid and all the forty samples were luted to the human teeth with resin luting cement. The samples were stored at 37<sup>0</sup> C for 24hrs in distilled water. The samples were tested for maximum load failure using **Universal Testing Machine**. The data obtained was analyzed statistically by **One Way ANOVA** and **Tukey HSD Test**.

Group D showed the highest mean bond strength of **22.451 ± 2.710 MPa** with a range between 17.87 MPa to 26.21 MPa. This was followed by Group C which showed the mean bond strength of **15.659 ± 3.569 MPa** with lowest and highest being 11.43 MPa and 20.58 MPa respectively. Group B resulted in the mean bond strength of **13.025 ± 1.618 MPa** with lowest and highest being 10.45 MPa and 15.40 MPa respectively. The lowest bond strength was recorded by Group A with the mean bond strength of **10.60 ± 1.384 MPa** with lowest and highest being 8.50 MPa and 13.14 MPa respectively.

One Way ANOVA Test for teeth sample shows significance of load value among all the pre-surface treatment at 5% of confidence level (p<0.05).

Future studies need to be done to evaluate the long term shear bond strength with other pre-surface treatment modalities like laser etching or pryosil-pen

technology. Further studies may also be done to evaluate the effect of these pre-surface treatments clinically to enhance the results obtained with the present study.

**Within the limitation of this study following conclusions was made:**

(1) There is **significance differences** exist in long-term durability to human teeth between with and without pre-surface treatment of porcelain veneers to human dental hard tissue.

(2) There was **significant difference** between APF gel/sandblasting/acid etching with HF.

(3) Although HF is considered to be a hazardous substance in causing various disorders and in spite of difficulty in storing HF, it is still considered best among various pre-surface treatments.



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